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An Engineering Study of the Effluent Disposal Problems of the Louisiana Raw Sugar Industry.

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AN ENGINEERING STUDY OF THE EFFLUENT DISPOSAL PROBLEMS OF THE LOUISIANA RAW SUGAR INDUSTRY

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ABSTRACT

Cane sugar manufacture is Louisiana's oldest industry, dating back to 1750. The cane grinding operation is seasonal, starting in mid-October and lasting about 70 days depending upon the crop and weather.

Cane juice, molasses, and sugar, because they are readily utilized by most biological organisms as food, are strong sources of organic pollution. Sugar and molasses have Biochemical Oxygen Demands of 1,250,000 and 930,000 ppm, respectively.

Although the method of processing sugar cane into sugar is the same as it has been for many years, changes in drainage patterns and factors have brought pollution problems created by the sugar industry into focus.

With the influx of other industries in the sugar area the populace which formerly accepted pollution resulting from sugar processing as a necessary burden, is now striving to keep the waters suitable for recreation.

The average throughput of Louisiana raw sugar factories has increased over the years as a result of low sugar prices and rising costs. Many factories now handle twice the amount of cane for which they were originally designed. This overloading

has increased pollution of the streams because of spills and/or entrainment in evaporators and vacuum pans.

Mechanized methods of cane harvesting have increased the amount of field mud delivered with the cane so much that over half of the mills now are obliged to wash it prior to processing. The effluent from the washing operation is heavily laden with sugars, soil, and pieces of broken cane.

Plant effluent surveys, made by the author, showed that the principal effluents not readily controlled by efficient factory operation are the cooling water from the barometric condensers and cane wash effluent. Factories were losing substantial quantities of sugar per day through entrainment. This problem was remedied by revising the entrainment removal devices on the evaporators and vacuum pans.

A study was then made of the cane wash effluent problem in order to effect a reduction in effluent volume while maintaining effective dirt removal from the cane and to develop an economical treatment.

It was shown that the volume could be reduced to one-third by multi-stage washing without danger to the factory from biological growths. Batch reactors were utilized to evaluate the effects of aeration, temperature, and nutrient addition on the bio-stabilization of cane wash effluent.

Aerobic operation was found to be several fold faster than anaerobic. The effect of temperature was found to closely follow the van't Hoff-Arrhenius relation, i.e., the rate approximately doubled for each 10°C increase in temperature. An analysis of variance showed that nitrogen addition in the ratio of about 1:20 nitrogen to B.O.D. was optimum for rapid stabilization. Potassium and phosphorous had no significant effects.

INTRODUCTION

The problems facing the Louisiana sugar industry in abating and preventing water pollution are as diversified as the industry itself. However, there is a single fundamental goal--the control of discharged wastes so that water quality is suitable for further use by others. This requires that adequate pollution control measures be devised and respected by each link in the chain of users of water from any given stream.

A sugar factory must have water for cooling, steam generation and for general utility purposes. It became imperative to find a way to overcome the pollution problem if the industry was to survive. In order to carry out this goal several raw sugar factories in Louisiana formed in 1954 the Louisiana Sugar Producers Waste Control Council. This organization made arrangements with the Chemical Engineering Department of Louisiana State University to sponsor the research project upon which this dissertation is based.

CHAPTER I

THE RAW SUGAR INDUSTRY IN LOUISIANA - POLLUTION PROBLEMS

The raw sugar industry was established in Louisiana[37] in 1750. During the formative years of the industry transportation in the state was mainly by water and the industry developed along major waterways. Raw sugar factories are found along the Mississippi River, Bayou Lafourche, and Bayou Teche. There is a heavy concentration of mills along Bayou Lafourche extending from Donaldsonville to Lockport and on Bayou Teche from Breaux Bridge to Franklin. In addition, there are factories scattered along various small bayous such as Erath Factory on Bayou Tigre and Meeker on Bayou Boeuf. Altogether there are about 50 in the state.

Raw cane sugar manufacture, like most agricultural industries, is seasonal. Factory operation extends from late October through December, a period of 60-70 days. The grinding season starts as soon as the sucrose content of the cane has reached a satisfactory level and ends when the crop is either completely harvested or is killed by freezing weather. This situation necessitates that the factories operate at maximum capacity, 24 hours a day, 7 days a week until the crop is harvested. While the number of operating factories has decreased through the years, the cane acreage has remained constant and the production of sugar is at an all time high. The result is that the remaining factories are accomplishing higher and higher throughputs each year. The industry has been a marginal one for many years and is unable to invest any considerable amount of money in plant improvements or in facilities for better usage of water.

The problem of water pollution by sugar factories is not new. References are found in the literature as far back as the later 19th century[4,8,11,21,32,38]. Many of the earlier investigators were handicapped by a lack of analyses which would provide them with numerical values rather than arbitrary comparisons of "good" and "bad" effluents. Most raw sugar factories today do not differ greatly in their operations from those of a quarter century ago. Although the mills then discharged large pollutional loadings the pollution problem was not as critical as it is at present.

Three principal factors have been responsible for the present effluent problem. First, and most important, is the obstruction to flow in the bayous by flood control structures. Erected by the U.S. Army Engineers as a result of the 1927 flood disaster, these structures have changed once flowing streams (particularly the Teche and Lafourche) into comparatively stagnant drainage outlets with little flow except during periods of heavy rainfall. In the Teche, for example, the flow is upstream as well as downstream, depending upon the prevailing wind, rain, and tidal conditions. Thus one often finds that waste discharged days previously has been carried back upstream to the door of the very mill that discharged it. At other times the level of the water may drop until much of the stream bottom is exposed. Figure 1 shows the level of Bayou Teche at daily intervals during the 1954 grinding season. It should be noted that the level during the latter part of the season, when the waste disposal capacity of the stream was most needed, was appreciably less than at the beginning.

The second factor in this problem is the increased throughput of the average mill. Figure 2 shows the hourly grinding rate of the average mill in Louisiana over a number of years. It shows a steady upward trend in grinding rate per factory. As labor and material costs continue to increase, the factories find that they must process more cane to stay on a sound economic footing. Those that cannot expand their operation find themselves operating at a deficit. The mills that continue to operate then receive the cane formerly processed in the extinct ones. Because sufficient profits have not been earned this steady increase in capacity has not been accompanied by a corresponding enlargement of plant facilities. Consequently,

VARIATION IN WATER LEVEL OF BAYOU TECHE

(As Read At Gauge Of Iberia Sugar Co-Op., New Iberia)

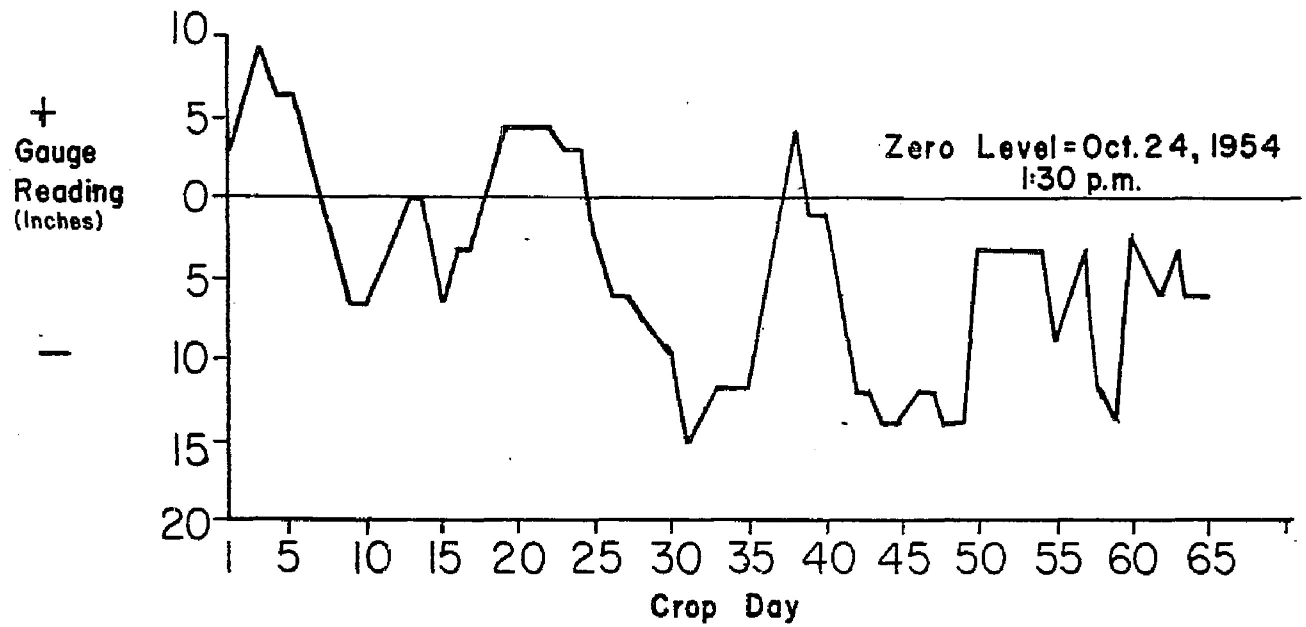


FIGURE 1

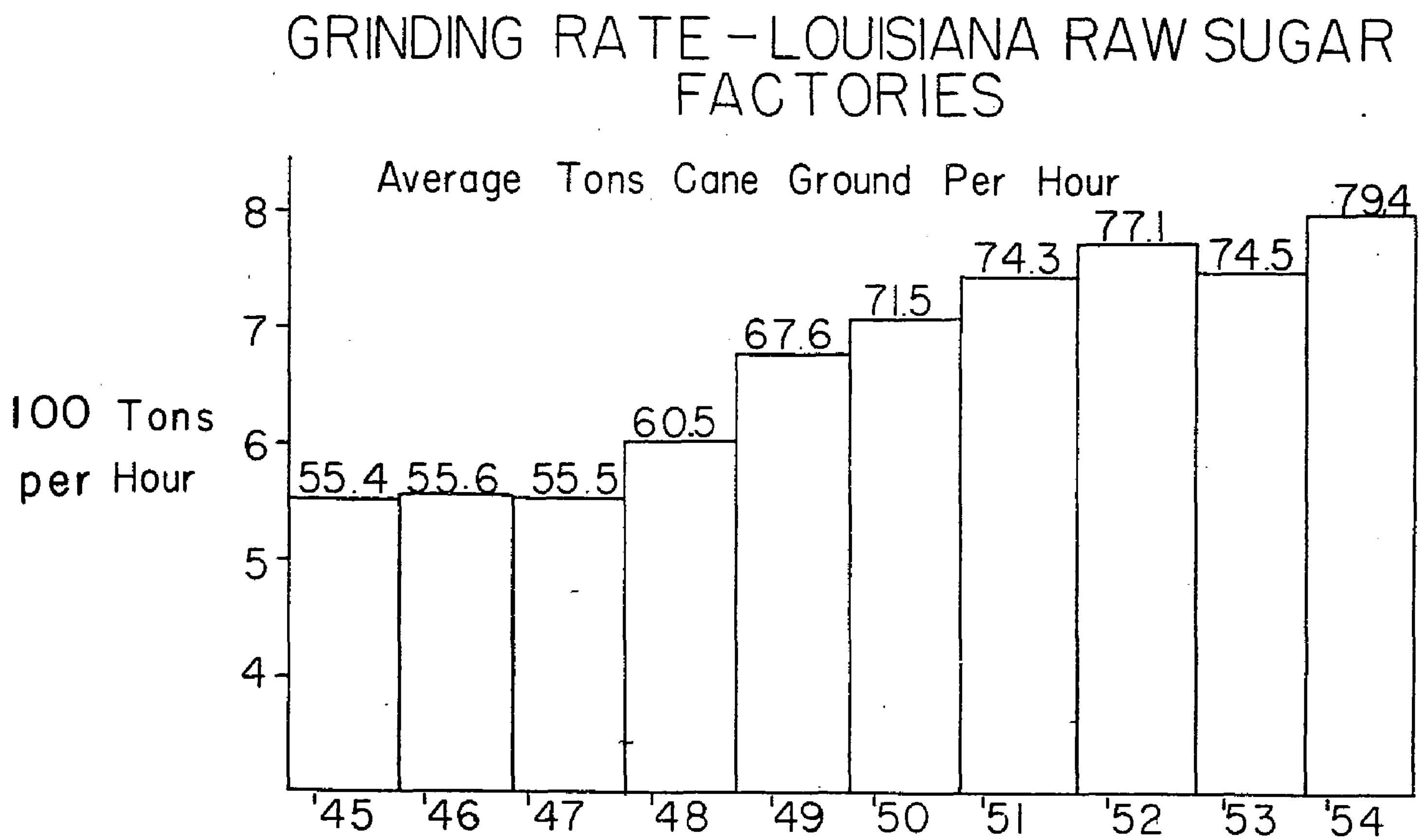

GRINDING RATE - LOUISIANA RAW SUGAR FACTORIES
Average Tons Cane Ground Per Hour
100 Tons per Hour
8
7
6
5
4
55.4
55.6
55.5
60.5
67.6
71.5
74.3
77.1
74.5
79.4
'45
'46
'47
'48
'49
'50
'51
'52
'53
'54

FIGURE 2

many of the mills are now operating at far greater rates than they were designed to handle. Overloading of equipment has caused the problems of spills and entrainment to become pollutionally important.

The third factor causing pollution problems for the sugar industry is the widespread practice of washing the cane entering the mills. This is done to remove loose dirt and trash from the cane before it is processed. Rising labor costs have forced cane growers to adopt mechanized methods for harvesting cane. This has increased the amount of foreign material contained in the cane delivered to the point where more than one-half of the raw sugar houses in Louisiana find themselves obliged to wash the cane as it enters the factory. The effluent from the cane washing operation is heavily laden with sugars and other organic materials which pollute the body of water that it is allowed to enter.

In past years, soon after the start of each cane grinding season, the bayous of South Louisiana have become badly polluted. Bayou Teche, for example, from New Iberia to Charenton has become septic and covered with a blanket of dead fish.
At one time the economy of the area was solely dependent upon the sugar industry and consequently the pollution was accepted by the majority of the people as a "necessary evil". In

recent years, however, the economy has shifted from sugar to oil. Many people are employed either directly in oil production or in supplying goods and services to those who are. This industrialization has brought about a higher standard of living and a desire for community self-improvement. Sportsmen's groups through the Louisiana Stream Control Commission have brought great pressure to bear upon the sugar industry to cease and desist from its pollution of public waters.

In many of these areas fresh water districts have been established to supply the population with drinking water. These are public organizations that are anxious to keep costs to a minimum. Since the cheapest source of fresh water is the bayou, these organizations prefer to use it. An example is a small town on the Teche. Formerly water was obtained through a pipe line about 25 miles long extending to another stream. This line deteriorated with time and rather than replace it the town started using water from nearby Bayou Teche. The city water, although chemically treated, has a very unpleasant taste. During the season when water hyacinths are dying, a characteristic bitter taste is present in the drinking water. The intake of the water treatment plant is only a few hundred feet from one of the largest sugar factories in the area. Like many other Louisiana sugar factories, it is in the unhappy position of being an established plant that suddenly has no place to dump its effluent.

CHAPTER II

THE OPERATION OF A RAW CANE SUGAR FACTORY

In order to present the full scope of this work it is advisable to first describe the operation of a sugar factory.

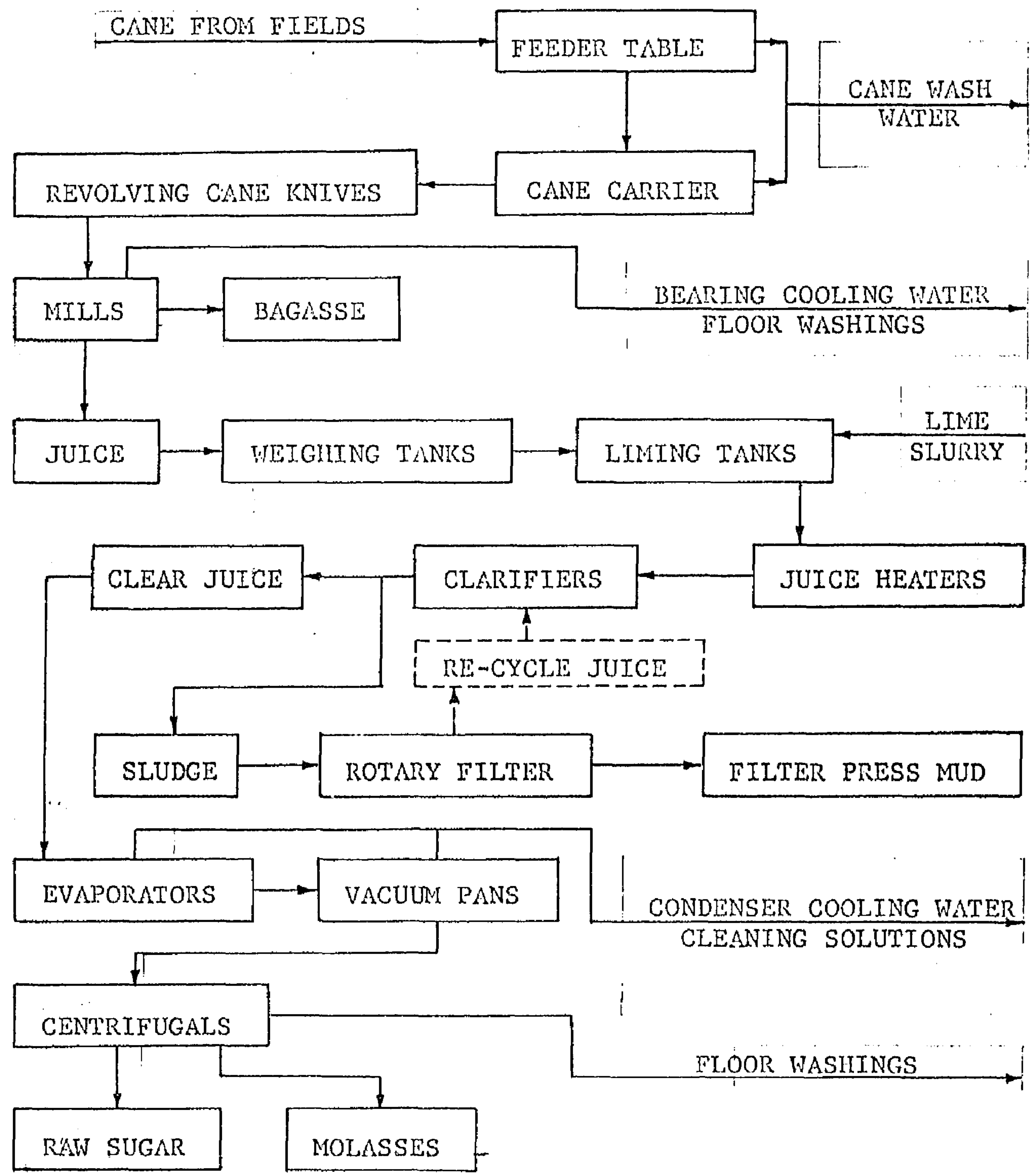
In the field, cane is cut by mechanical harvesters which sever it at the bottom, trim off the top and pile it in rows. After burning to remove excess leaves the cane is loaded into trucks by mechanical grabs. A bundle weighs 2-3 tons. Each truck carried about 20 tons of cane to the factory where the bundles are unloaded by crane and placed on a conveyor known to the industry as a "feeder table". Here the cane is often washed by water sprays to remove adhering dirt and trash. From the feeder table the cane drops to a slat conveyor (where it may be washed also) and is transported through one or more sets of revolving knives which chop it into small pieces. The juice is squeezed from the cane by multiple-roller mills. The woody material remaining (Bagasse) may be burned as fuel, sold as animal litter, or baled and sold for the manufacture of wall board (Celotex). Juice from the mills is screened, weighed, treated with lime to a pH of about 8 and heated by steam to about 210°F in a shell-and-tube heater. The juice then passes into insulated clarifiers where the suspended solids are removed by settling. Underflow (mud) from the clarifier is filtered and the filtrate re-circulated through the process. The filter cake is either mixed with water and pumped as a slurry into impounding basins or hauled to nearby fields to serve as fertilizer. Clarified juice is passed through multiple-effect evaporators where water is removed to concentrate it to about 60% solids (syrup). The syrup is further concentrated in vacuum pans, where sugar is crystallized from the mother liquor. The mixture of liquid and sugar crystals is dropped into a perforate basket, vertical centrifugal where the crystals are separated. The liquid from the centrifugals is re-concentrated and another crop of crystals is obtained. Generally three "strikes" or batches of crystals are obtained from a given syrup. The raw sugar is usually about 97% sucrose and is sold as such to refiners. Spent liquor or final molasses may be sold for cattle feed, fermentation into alcohol, or yeast production.

Spencer and Meade[37] give a detailed discussion of each step of sugar manufacture.

The general flows of a raw cane sugar factory are shown in Figure 3. In this figure the major effluents are shown as being discharged to the right of the diagram.

FIGURE 3

PROCESS FLOW DIAGRAM OF A CANE-SUGAR FACTORY
SHOWING DISCHARGED EFFLUENT



CHAPTER III

THE UTILIZATION OF WATER IN RAW CANE SUGAR MANUFACTURE

Water requirements for raw cane sugar manufacture may be grouped into two general categories:

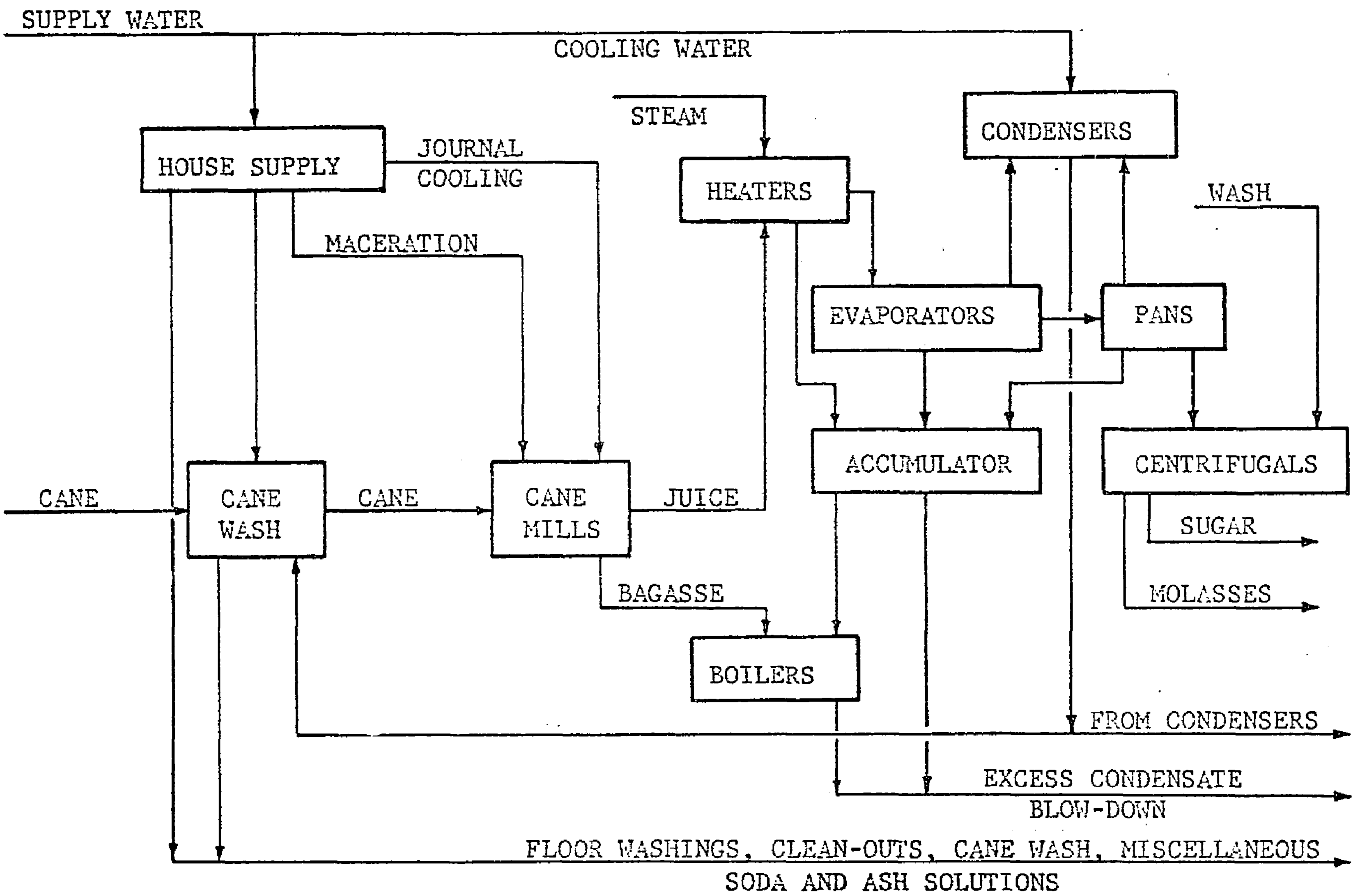
(a) Water used for cooling purposes
(b) Water used in processing

The utilization of this water is best visualized by referring to Figure 4, "Water Utilization Diagram of a Cane Sugar Factory". Water for cooling purposes is used in the barometric condensers of multiple-effect evaporators and vacuum pans, and to cool the sugar massecuites in crystallizers. The quantity of cooling water used varies from 3,000 to 12,000 G.P.M., depending upon the size and equipment of each factory. It has been estimated by Keller[17] that the Louisiana industry as a whole uses about 360 million gallons of water per day during the grinding season. The water generally comes from streams or wells near the factories and is discharged to a stream or adjacent swamp area. Many mills utilize closed re-circulating systems with spray ponds to reduce the amount of water needed from outside sources.

Water is actually used in the process for: (1) washing sugar crystals, (2) boiler feed water, (3) coolant for machinery bearings, (4) spraying onto the ground cane that has previously passed through one or more sets of rollers to aid in dissolving

FIGURE 4

RAW CANE SUGAR FACTORY
WATER UTILIZATION FLOW DIAGRAM



sugars so they may be removed in subsequent sets of rollers (maceration), (5) sanitary and clean-up purposes around the factory, and (6) washing cane as it enters the factory to remove mud and trash. Generally, the condensate recovered from the multiple-effect evaporators is more than ample to supply the needs for boiler feed water, maceration, and washing sugar. About 75% of the water in the juice is removed in the evaporators and all but the vapors from the last (usually third) effect are recovered. The vapors from the last effect are condensed by direct contact with cooling water in barometric type condensers and are lost so far as condensate recovery is concerned. In factories that wash cane, warm water from the condenser tail pipes is utilized in part. Any excess condenser water is discharged to a nearby stream. Cooling water for bearings is generally fresh domestic or well water. It picks up traces of grease or sugars and, although usually not a bad offender, is generally impounded in detention basins. Water for sanitary and clean-up purposes is generally domestic water. However, water from the stream is sometimes utilized where treated water is unavailable.

Many factories mix the filter-cake with water and pump the resulting slurry to detention ponds. The material is allowed to remain there for several months to decompose and dry out by evaporation. During the summer months the dried material is removed and spread on the fields as fertilizer. Formerly this material was dumped directly into a stream, a practice which is no longer continued except by some factories along the Mississippi River where large dilution is available. As the sugar mill effluent is not toxic, the organic material therein is readily purified by biological stabilization in the river.

In order to understand the pollution problems of the sugar industry a knowledge of the constitution of the waste discharged and the manner in which it exerts a pollutional effect upon a body of water is desirable. Sugar factory wastes are similar to those from other food processing, starch manufacture, pulp, and paper mills in that they pollute the waters by exerting an "oxygen demand" by virtue of their utilization as food for micro-organisms present in the environment. This is in sharp contrast to the "toxic" pollution exerted by the wastes from certain industrial plants which exerts itself in the form of poisonous material in the effluent. Examples of this toxic type of pollution are cyanide wastes from plating plants, acids found in coal mine drainage and the waste "pickling" liquors of the steel industry. A noteworthy example of these are certain phenolic materials which have been discharged into the Mississippi River in the past as constituents of oil refinery effluents in the Baton Rouge area. When chlorinated in domestic water treatment plants these wastes form chloro-phenols which caused the water in the city of New Orleans to have a "medicinal" taste in concentrations as low as 1 part per million. In 1918 serious complaints of intolerable "medicinal" tastes in the water were registered in Milwaukee. The source was found to be a phenol plant on the Indiana side of the lake[28].

Thus, the sugar mill pollution problem is one of organic material entering the body of water where it acts as food for micro-organisms present there. It is the growth and reproduction of these organisms with their accompanying action upon the surrounding atmosphere, water, plant and animal life which is the undesirable end pollutional effect. If the material remained sterile after it entered the water there would be no pollutional effect. However, it is this same microbial action which allows the earth to continually purify itself and replenish life over and over again through the ages. Without it the world would be a barren refuse heap.

A detailed discussion of the mechanism or biological decomposition will be found in texts on waste[15,28,33,36] treatment. Biological activity uses up oxygen from the water. When the surroundings contain ample dissolved oxygen to supply the needs of stabilization of the effluent the decomposition is said to be "aerobic". When dissolved oxygen is not present, the organisms utilize combined forms of oxygen or oxidizing compounds. This "anaerobic" decomposition produces hydrogen sulfide, methane, and numerous intermediate products which are foul to the taste and smell as well as being toxic.

The immediate and most striking result of oxygen depletion in a stream is the death of fish. When the dissolved oxygen concentration begins to drop, fish begin to die. The game fish are killed first and as the oxygen content of the water approaches nil the hardy "outlaw" fish such as gar are overcome.

Fish will try to leave a location where the dissolved oxygen is depleted, but frequently they are unable to escape. It was common to see Bayou Teche in the vicinity of Jeanerette, where there are four sugar factories, covered by fish floating dead on the water after the mills began operation.

Organic pollution fosters the growth of innumerable types of plants and organisms which utilize the material for nutrition and growth. One might visualize this complex as a thriving community wherein a source of new money is the organic pollution which is used many times as it passes from person to person, each obtaining a living and in turn helping to support others. This interrelationship is excellently discussed by Phelps[28].

The effect of this micro-organism community is diverse. Often foul odors and tastes result which render the water unfit for drinking. Unpleasant smells make the neighborhood undesirable for habitation, a condition which is very unfavorable to community development. Grossly polluted waters develop a black color and become opaque due to the micro-organism life therein.

It is a known fact that polluted waters harbor pathogenic bacteria. In the past, many epidemics of typhoid fever and dysentery have been caused by polluted drinking water. Conditions which favor the growth of micro-organisms, namely a source of organic enrichment, will conserve and promote the life of pathogenic bacteria[14].

A number of tests have been developed to evaluate the magnitude of water polution. The ones which are of importance to this study are the D.O., O.C. and B.O.D. tests. A brief description of these will be found in the appendix.

CHAPTER IV

FIELD STUDIES

A survey of the literature showed that no comprehensive study had been made of commercially operating raw cane sugar factories. However the problem was recognized as far back as 1914 when it was estimated that 3000 pounds of sugar per million pounds manufactured were lost[26]. It was observed at this early date that "The most important source of pollution in Louisiana is that from sugar factories." The pollutional effects cited were removal of oxygen from streams, death of fish, and the production of unpleasant odors.

In 1944 a study was made by the Louisiana Stream Control Commission on the effluents from the Audubon Sugar Factory, an experimental sugar mill which is operated by Louisiana State University[19]. Although other work prevented a detailed study, a series of tests and flow measurements was carried out in order to determine the strengths of typical sugar factory wastes. The experimental sugar factory ground cane at the rate of 10 tons per hour for only 11 hours per day, 5 days a week, as compared to a typical commercial operation of 80 tons per hour, 24 hours a day, 7 days a week. Its wastes in contrast to a full size plant were very small. The Audubon factory is an ideal operation which is supervised by the Department of Chemical Engineering. The survey showed this to be true by pointing out that the B.O.D. of the Audubon condenser water was only 21 compared to 162 for an unnamed commercial factory grinding 1300 tons of cane per day and 37 for a second factory grinding 2700 tons. The report stated that the equivalent population required to produce this effect would be 1154 people for Audubon and 48,600 and 30,000 for the two commercial factories respectively.

Another source of pollution pointed out in the Audubon survey was the wastes resulting during bi-weekly cleanings. The heat transfer surfaces of evaporators and heat exchangers are cleaned periodically using caustic soda and muriatic acid alternately. The small volumes of these wastes made them much less a problem than the day to day effluents, even though these wastes are relatively concentrated.

The results of the Audubon tests were cited by Cusachs[4] at the 1947 Short Course for Water and Sewage Plant Operators held at L.S.U. The findings are summarized in Table I.

As a result of this survey it was concluded by the Stream Control Commission that all sugar mill operators in the state should discharge only their condenser water into public streams. It was also stressed that the population equivalent of the two commercial factory effluents was greater than the total population of all communities along their receiving

TABLE I

EFFLUENTS PRODUCED AT THE AUDUBON SUGAR FACTORY - 1944*

Wastes Produced Daily

Waste	Quantity	5-day B.O.D.** p.p.m.	Pounds B.O.D. / Pound Cane
Mill Wash Water (Floor sweepings)	450 gal/day	17,500	0.55
Filter Solids	1856 #/day	206,000	3.23
Filter Cloth Wash Water	225 gal/day	26,000	0.41
Condenser Water	792,000 gal/day	21	1.17
Total			5.36

Bi-weekly Cleaning Wastes***

Waste	pH	Gals/Day	5-day B.O.D. p.p.m.	Pounds B.O.D. / Pounds Cane
Evaporator Wash Water	7.3	150	1480	0.0028
Caustic Rinse Water	10.2	450	82	0.0005
Acid Waste Water	1.3	860	1050	0.0120
Acid Rinse Water	2.2	450	200	0.0012
Total				0.0165

* Survey made by Louisiana Dept. of Wildlife and Fisheries

** Refers to B.O.D. pick-up. Actual B.O.D. of circulating cooling water was 139.

*** Quantities are for the day of factory cleaning only.

streams.

Very little further attention was given the cane sugar factory pollution problem until 1954. At that time the Louisiana Stream Control Commission set a discharge limit of 1-1/2 pounds of B.O.D. per ton of cane ground on those mills discharging into Bayou Teche. The restrictive orders, issued with the intent of improving the condition of Bayou Teche, were unsuccessful. Several factories were served by the Stream Control Commission with "Cease and Desist" orders. At this time a group of factories banded together and formed the Louisiana Sugar Producers Waste Control Council, the originator and sponsor of this project.

In-Plant Effluent Studies

Prior to each grinding season inspection visits were made by the author to the factories that were participating in the project. There were eight factories in the original group. Sampling and analytical procedures were provided for each mill and detailed instructions given to their chemists and other supervisory personnel involved.

The factories purchased and set up the necessary equipment to perform O.C. and D.O. analyses. Because a rather expensive constant-temperature incubator is needed to perform B.O.D. analyses they were run only at L.S.U. on samples which were delivered there at frequent intervals. Visits were made to the factories at intervals of one week or less. At the time of each visit the operation was inspected from a pollution abatement standpoint, the laboratory analyses performed at the mill were examined, recommendations were made to operating personnel to alleviate any trouble found and samples were collected for immediate transport to Baton Rouge. These samples were packed in ice and rushed by auto or by private aircraft to L.S.U. in order to prevent any deterioration prior to analysis. In addition to B.O.D. analysis, the O.C. was determined on most samples to obtain a correlation between the two so that the samples analyzed by the factory laboratories could be used in estimating B.O.D. pollutional loadings. This correlation is shown in the appendix.

Weirs were installed at the mills to measure flows. Readings were obtained from them at regular intervals throughout the grinding season. In addition a set of three Parshall flumes having throat widths of 3", 6", and 3' were constructed and a "Brown" recording and integrating flowmeter suitable for these units was purchased. This equipment made it possible to obtain instantaneous flow rates, total flows, and a profile record of effluent discharge volumes throughout the day from a given source. When no flow measuring device was available, flows were estimated using the characteristic curves for the pumps involved.

Analytical reagents required to run the O.C. and D.O. tests were prepared for the factories in order to insure reliable results. Nearly 100 gallons of standard solutions were supplied to the factories each year.

The results obtained from the first survey made it evident that there were certain key sources of pollution common to most factories. Efforts were then made to have the factories take the necessary correctional steps. This was not difficult as it was shown that much of the pollution was being caused by material that should be leaving as product.

A survey of the factories during the next season showed rather striking improvements resulting from various steps taken as a result of the initial survey.

Figure 5 shows the B.O.D. values for six factories before the 1954 season, during the season, and during the following season when remedial measures advocated as a result of the initial survey had been put into effect. In order to protect the confidential nature of the survey the factories are identified by letter and general location. It is of interest to note the rather wide variation found from factory to factory.

Figure 6 shows the pounds per day of B.O.D. discharged

FIGURE 5

AVERAGE VALUES OF B.O.D. IN BAYOUS
AT VARIOUS SUGAR FACTORIES

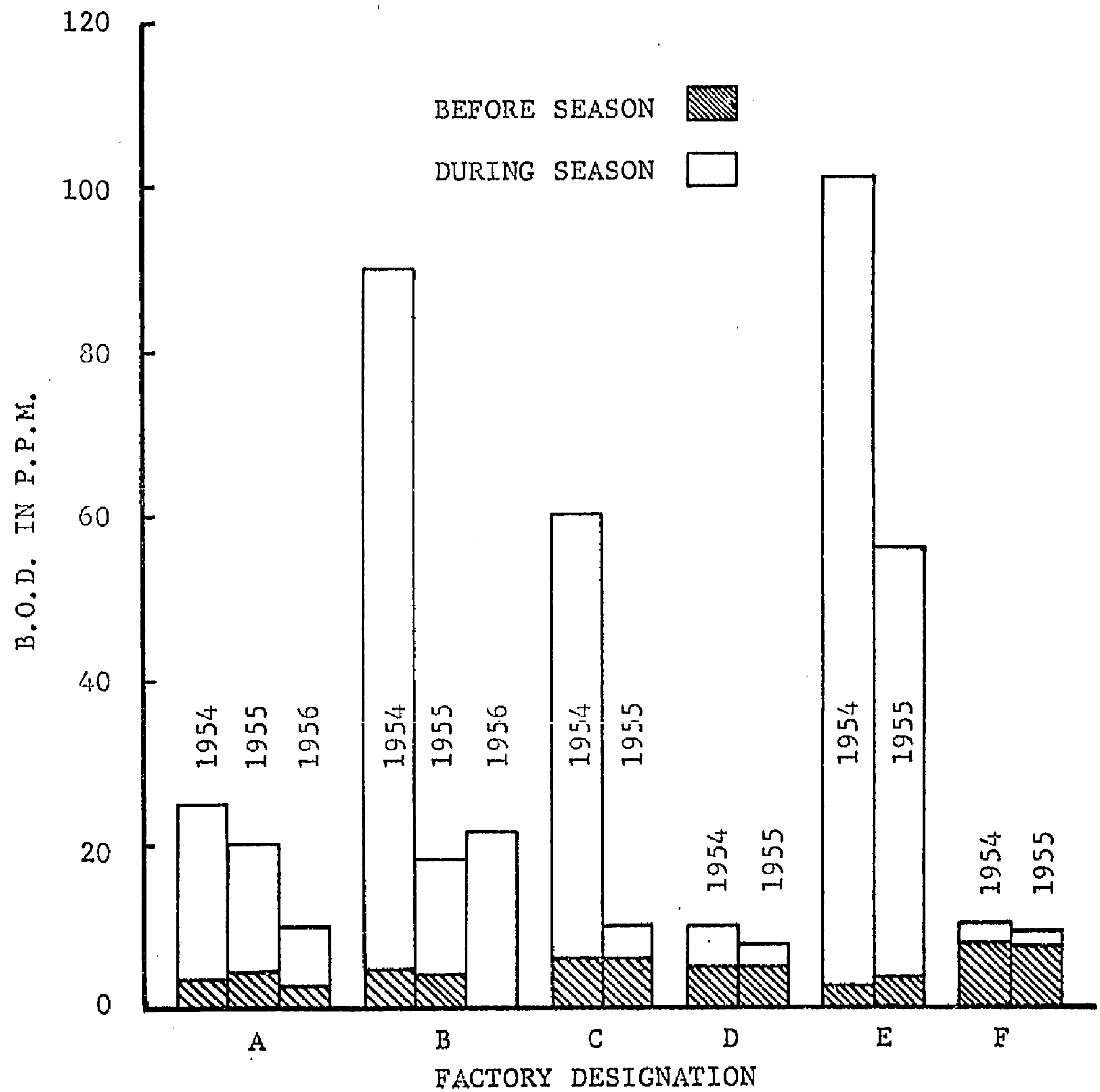
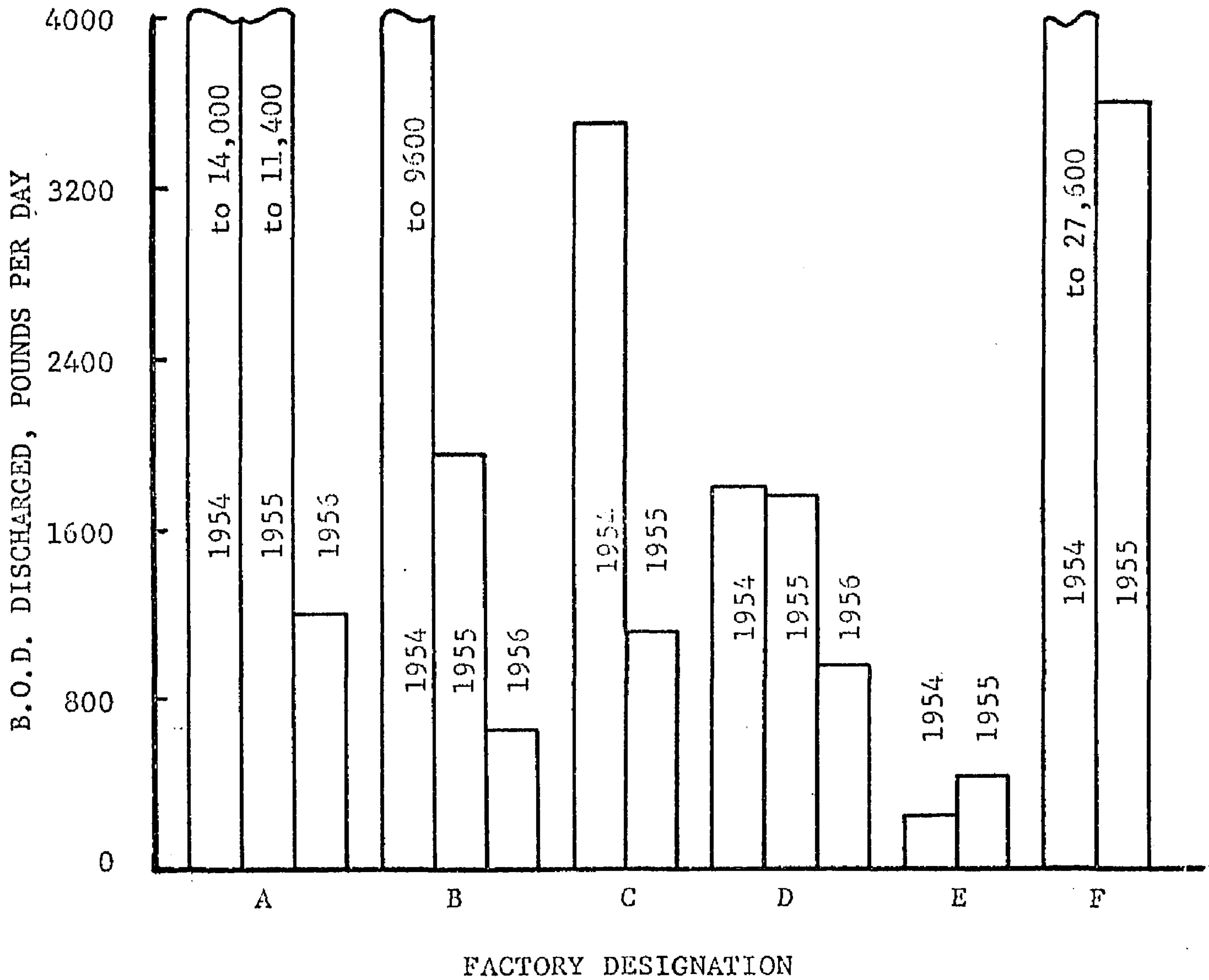


FIGURE 6

B.O.D. DISCHARGED INTO BAYOUS
AT VARIOUS SUGAR FACTORIES



by several factories. It is a key feature of the survey since the other pollutional effects upon the stream are a result of the B.O.D. discharged.

Figure 7 shows the dissolved oxygen in Bayou Teche at each of the six mills both before and during the grinding seasons. Note that during the 1954 season four of the six locations experienced average D.O. concentrations below the minimum of 2 p.p.m. recommended for aquatic life. There were fish kills at all four locations.

Figure 8 shows the average temperatures present in the streams at the locations studied. There was little variation from factory to factory in the same stream and not much difference between any of the locations. Note that the average for all factories studied was about 19 degrees Centigrade which is considered an excellent cooling water temperature by most industrial plants.

<u>Raw Sugar Factory Effluents</u>

A consideration of the waste disposal problem of a cane sugar mill quickly brings the realization that there is not one, but many problems involved. Several distinctly different types of wastes emanate from each mill and each of these presents a slightly different disposal problem. These wastes will now be considered separately.

FIGURE 7

AVERAGE VALUES OF D.O. IN BAYOUS AT
VARIOUS SUGAR MILLS

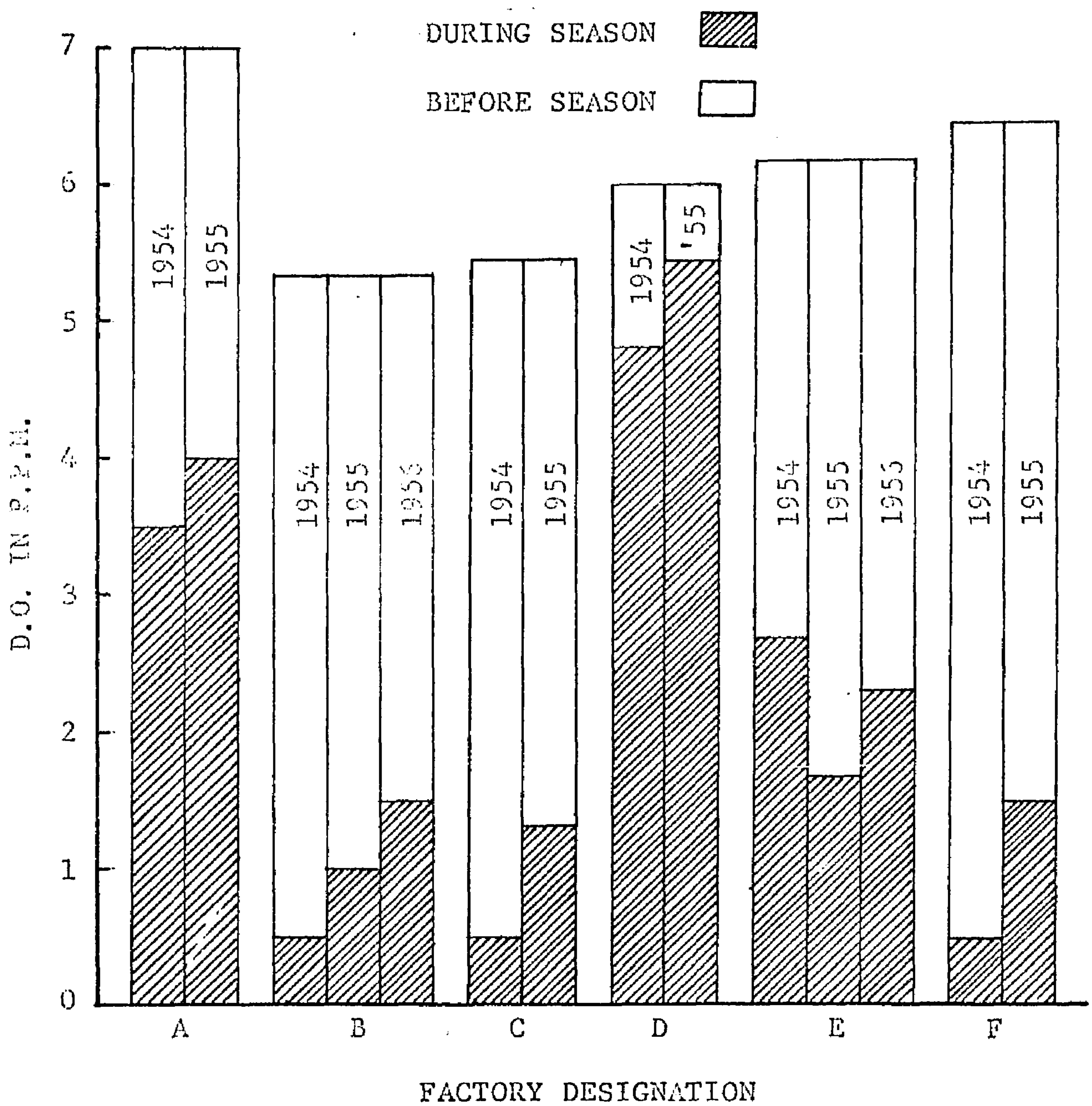
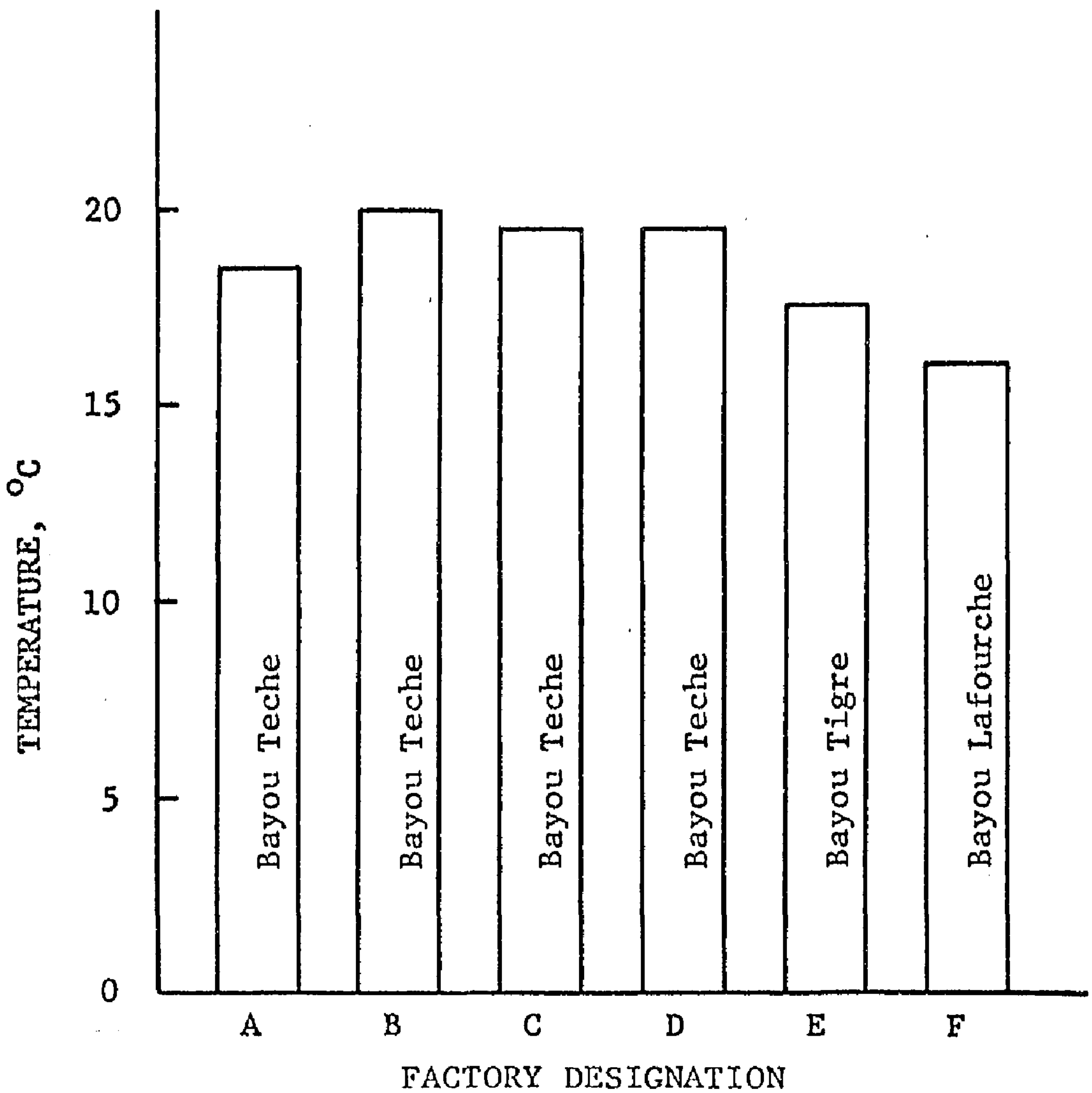


FIGURE 8

AVERAGE VALUES OF TEMPERATURES IN BAYOUS AT VARIOUS SUGAR MILLS



Condenser Cooling Water

By far the major amount of water used in cane sugar manufacture is that used for cooling the barometric condensers used in connection with the operation of evaporators and vacuum pans. Theoretically, there is no reason why we should not be able to operate vacuum pans and evaporators successfully without any entrainment of sugar or other contaminating materials in the condenser discharge. With respect to entrainment, the main offender is the last effect of the multiple-effect evaporators employed in every raw cane sugar factory. Here, because of the low pressure, the vapor velocities are higher than in the preceding effects and the entrainment is more critical.

Entrainment is generally due to one of three causes: (1) working the plant too fast, (2) incorrect operation, and (3) improperly designed equipment. In Louisiana there has been a steady increase in grinding rates which has imposed a heavier loading upon the evaporators and vacuum pans, making them very susceptible to entrainment. Actually, entrainment results from the carry-over of small drops of liquid by the vapors leaving the unit. Vapor bubbles form in the liquor in the tubes and as they rise they increase in size and velocity. When the bubbles reach the surface they burst and shoot droplets into the vapor space above at velocities of up to 25 feet per second in the low-pressure last effect. If the boiling rate is excessive the mixture of liquid and vapor will shoot out of the tube bundle with sufficient speed to carry large droplets directly into the vapor outlet. In addition, many of these droplets will be atomized and entrained in the rapidly moving vapors. Many of the droplets entrained in the vapor by the bursting bubbles will be so small as to form a mist that is not susceptible to the action of gravity or centrifugal force to any large degree. This form of entrainment is not efficiently removed by the usual baffle-type separators found in most sugar factories. The tendency toward entrainment increases with a decrease in pressure from the first to the last effect. In the last body the resistance to upward movement is less because of lower vapor density, therefore the small droplets are projected to a greater height.

The most common operational errors are incorrect liquid level and variations in vacuum within the evaporating unit. Many operators believe that the tube bundle should be completely covered with liquid to provide maximum contact with the heat transfer surfaces. However, experience indicates that the most efficient use is obtained when the liquid height is about 1/3 of the tube bundle length. Under these conditions a regular convection flow will exist. If the level is raised there will be turbulent upheavals resulting in increased entrainment.

The vacuum in a unit may be subject to variations from two causes. If several units are connected to a single condenser changes in one unit will cause the pressure in the others to vary. Similarly, if a single pump supplies water to several condensers, variations in flow through one condenser will affect the others. Since the pressure determines the boiling point in the vessels, any variations of it will result in alternate periods of no boiling followed by intense flashing. During the flashing period the contents of the unit may froth up into the separators. In such a case the separators are ineffective.

Improperly designed units generally are of two classes; those having improper liquid inlet distributors or those having an irregular heat distribution. A flashing effect takes place when a liquid flows at its boiling point from one vessel into another which is at a lower pressure. This is the result of the lowered boiling point which causes the excess heat content of the inflowing liquid to be lost by vaporization. The volume of vapor given off in each effect by flashing increases with the vacuum. In the last effect the vapor volume is about 700 times as great as that of the same weight of liquid. This large change in volume explains the turbulence that the flashing produces. Since the flashing occurs as the liquid enters the unit its effect can be minimized by proper feed distribution. Improper distribution of heating steam and inadequate venting of non-condensibles from the steam section of the heating bundles will result in irregular boiling. This will result in points of high turbulence within the evaporator causing considerable splashing and spray.

Every operator is attempting to get the most from his equipment. Frequently mills grind twice the amount of cane for which their equipment was originally designed. For this reason it is of prime importance to every mill in the state to have proper entrainment removal devices and to give them the maintenance they need. The only way in which entrainment can be eliminated is by the installation in the evaporator or vacuum pan of adequate, and properly designed, separators or "catch-alls". These are available in numerous designs from various fabricators who specialize in this type of equipment. In most factories, in addition to eliminating a large portion of the pollution problem, the installation of such a separator will normally result in increased yields of sugar sufficient to pay for its installation cost in one or two grinding seasons.

At the start of this research program persons having supervisory responsibility for the participating mills were interviewed. None of the eight mills in the original sponsoring group would acknowledge having any entrainment difficulty at that time. During the first season of the program it was found that six of the eight mills were having heavy sugar losses through entrainment. The source of this trouble was traced in each case to the last effect of the multiple-effect evaporators. The B.O.D. of the combined condenser cooling water effluent for evaporators and vacuum pans varied from 50 to 400 parts per million. Flows were in the order of from 2,000 to 6,000 gallons per minute.

Based on the market price of sugar at that time and the fact that sugar has a B.O.D. of 1,250,000 parts per million it was calculated that the entrainment was costing the mills anywhere from $100 to $2,500 per day. These estimates, subsequently confirmed by low yields on the part of the mills, resulted in a notable increase in cooperation and interest from the mill owners. Examination of the entrainment separators in the factories showed that every mill having trouble was in such a position because of inadequate or mal-functioning entrainment removal equipment.

There were three principal shortcomings, one or more of which was found to be the cause of the equipment failure:

1. Separator return lines were too small to prevent caking and clogging by syrup returning to the unit.

2. Short-circuiting of separator due to lower pressure drop through the liquid return lines than through the separator proper.

3. Interior baffles corroded away due to improper maintenance.

Where catchall return lines are too small, syrup gradually cakes inside and a slug of sugar combined with bits of rust eventually seals up the passage. With the drain lines plugged the level of syrup which is deposited in the catchall gradually rises until it reaches the barometric condenser. Once this happens a steady flow of sugar from the unit into the cooling water outlet results.

Many of the return lines were designed in such a manner that the pressure drop through them is less than that through the separator itself. Because of this the vapor flows out through the return line and prevents separated syrup from returning to the body of the evaporator. Having no place to go, the liquid floods the separator and passes out through the vapor line into the cooling water and subsequently into the bayou itself.

Once the faulty separator installations were put in order, it was found possible to reduce the B.O.D. pick up of the cooling water from a value which may be as high as 500 parts per million to 5-15 parts per million. About 5 parts per million of residual B.O.D. pick up is believed due to non-condensables such as ammonia which come from the cane juice and are absorbed in the cooling water.

The seriousness of the entrainment problem among the eight member mills can be pointed up by citing a few of the figures found during the first (1954) grinding season. These figures represent the maximum, or worst condition found and not the average condition. Of the eight mills, three in the group are of relatively new construction and, in consequence, their separators are more properly designed possibly than those in the other five mills. In these three mills the B.O.D. pickup in the water passing through the condensers was, respectively, 15, 40, and 50 parts per million. On the other hand, among the five older mills of the group, the B.O.D. pickup of condenser water varied from 100 to as high as 360 parts per million with the average being in the order of about 180 parts per million.

Tests were occasionally made of the condenser water by mill personnel using the Alpha-naphthol test for sugars. It was found in many cases that a sample gave no test and yet was found to have a B.O.D. of over 100 parts per million. The Alpha-naphthol test was supposed to indicate concentrations of sugar as low as one part per million by weight. It has been difficult to convince sugar factory personnel that their time honored test is inadequate. Tests reported by the Government Sugar Experiment Station, Tainan, Formosa, indicate that Ammonia, Acetone, Nitrates, SeO_2 or Ferric Salts reduce the sensitivity of the Alpha-naphthol test.

A study of the Alpha-naphthol test was made to determine its sensitivity. It was found that fresh, clean solutions should be used. After a period of about one month the sensitivity of the reagent was found to diminish. Various light sources were tried and it was found that a yellow fluorescent light gave the best conditions for the test. It took as long as three minutes for the test color to appear using solutions a month or so old.

Various combinations of sugar solutions, Alpha-naphthol and acid were used to determine the best ratio for the test. The amount of acid used was found not to affect the test so long as an ample amount was present to establish two layers. With about 15 milliliters of solution being tested 8-12 drops reagent, and 5-10 milliliters of acid were sufficient to conduct the test at optimum sensitivity. Generally it was found that the greatest dependable sensitivity was 10 parts per million instead of the previously cited value of one part per million. The procedure for running this test is to be found in the appendix.

Soda and Acid: Caustic Soda (NaOH) and Hydrochloric Acid (HCl) are used to keep clean the heat transfer surfaces in heaters, evaporators and vacuum pans. This is done at intervals of 2 to 3 weeks throughout the season. The acid and soda are generally mixed in an impounding pit where they tend to neutralize each other. However, there is still a degree of basic character and therefore they are not released until the rainy season when there is sufficient dilution to permit their assimilation into the stream without any ill effects. In no case was this effluent found to be entering the streams and so it will not be further considered.

Filter Mud: Filter mud was once pumped into the streams in the form of a slurry. However, this practice was ruled out by state authorities when the first sugar factory problem came under investigation. This material contains 2-4% sugar which gives it a B.O.D. of at least 2500 parts per million. In no case was the mud found to be entering a stream. There were two general methods used for its disposal; pumping it as a slurry into a "mud-pond" where it is allowed to ferment and dry for several months after the grinding season has ended, or hauling the moist cake away by truck or cart. In either case the material ends up being spread over the fields as fertilizer. Many factories have special tractor carts which contain a screw conveyor to continuously spread the mud along the rows as it is driven through a field to be fertilized.

Floor Sweepings: Leakage of juice around the mills, juice tanks, juice pumps, and similar points can be a serious source of pollution. Spillage of juice, syrup, molasses, and sugar on the floors and premises of the factory can also be a source of pollution when the areas are washed down during routine cleanup or following rains which dissolve and flush out this material. Floor sweepings are generally highly pollutional and their B.O.D. is often in the range of 500-2000 parts per million. Since sugar has a B.O.D. of 1,250,000 parts per million and molasses, 930,000 parts per million, it takes very little "sweet water" to cause appreciable pollution trouble[38]. A serious problem in most of the older factories is the lack of definite knowledge as to the location of floor drains and sewers. There is also the problem of separation of the contents of some floor drains which carry contaminated water. All floor sweepings should be, and presently are, impounded in pits prepared for the purpose. To prevent overloading these facilities, the contaminated material should be separated from the clean effluent which may be returned to the bayou.

Cane Wash Water: A pollution difficulty encountered only by those mills which are troubled by muddy cane is that of disposing of the effluent resulting from washing cane. This effluent is fast becoming the number one pollution problem of the Louisiana sugar industry. Roughly half of the mills in the state now wash their cane, some intermittently, others, full-time. The effluent from this operation has a B.O.D. which ranges from about 200 to 800 parts per million depending upon the relative quantities of cane and water involved. Its volume is generally between 800-2000 GPM. Data obtained during the 1955 season showed that the loading varied from 1.2 to 5.5 pounds of B.O.D. per ton of cane. The physical characteristics of typical samples of cane wash water are shown in Table II.

The practice of washing cane evolved in recent years to combat large amounts of mud which are gathered from the field along with the cane itself. This is due in part to increased mechanization in cutting cane and bringing it to the mill. Much of the mud is received at the mill and weighed with the

TABLE II

PHYSICAL CHARACTERISTICS OF CANE WASH WATER

Analysis	Iberia		St. Marys	Sterling
Date	12/10	12/12	12/10	12/12
pH	6.7	7.2	7.3	6.8
Settleable Solids, ml/L	5.0	2.7	10.0	5.0
Total solids, ppm	3,827	2,012	3,138	2,625
Volatile, ppm	940	608	740	977
Volatile, %	24.6	30.3	23.6	37.2
Dissolved Solids, ppm	1,013	449	692	1,031
Volatile, ppm	712	319	276	726
Volatile, %	70.3	71.1	40.0	70.4
Suspended Solids, ppm (by difference)	2,814	1,563	2,446	1,594
Volatile, ppm	228	289	464	251
Volatile, %	8.12	18.5	19.0	15.7
Non-Settleable, ppm	1,659	773	1,113	1,170
Volatile, ppm	740	359	447	725
Volatile, %	44.7	46.4	40.1	62.0
Settleable Solids, ppm (by difference)	2,168	1,239	2.025	1,455
% of total	51.7	61.6	64.5	55.7
% of suspended	77.2	79.2	82.8	91.4
Volatile	200	249	293	252
Volatile, %	9.2	20.1	14.6	15.8

CENTRIFUGED CANE WASH WATER

Analysis	Iberia, 12/10	St. Marys, 12/10
Total Solids, ppm	836	628
% of total in effluent	21.8	20.0
% removed	78.2	80.0

TABLE II
(Continued)

CANE WASH INFLUENT

Analysis	Iberia, 12/10	Sterling, 12/10
pH	6.5	7.1
Total Solids, ppm	351	630
Volatile, ppm	170	367
Volatile, %	48.5	58.3
Dissolved Solids, ppm	257	549
Volatile, ppm	174	331
Volatile, %	67.8	60.4
Suspended Solids, ppm (by difference)	94	81
Volatile, ppm	0	36
Volatile, %	0	44.5

the cane. Although a sample of each load of cane is taken, and a deduction in the gross weight made to compensate for the trash found, the sampling procedure is such that the deduction is inadequate. For this reason, it is to the farmers' advantage to haul mud with the cane up to the point of the mills refusing to accept it. The increasing amounts of mud being brought in with the cane forces more and more mill operators to wash in order to stay in business. This tendency is shown by Gilmore[18] who states that half of the mills in the state of Louisiana wash cane. Many of those which do not wash regularly set up temporary washing apparatus such as a fire hose during times when the cane is muddy.

The savings resulting from cane washing come from increased sugar yield and from better operation that results throughout the plant. The entire chain of benefits is based on one factor, mud removal. It has been found that washing cane will reduce the amount of filter press mud as much as 100 pounds per ton of cane ground. The average mill in Louisiana ground approximately 2000 tons of cane per day in 1958 and the mud from this operation averaged about 4% sugars. Based on removing the mud before it enters the mill and thereby removing cane sucrose in the mills which might be lost in mud, washing saves roughly $480 per day of operation. By washing, the average mill can decrease its loss by about $0.24 per ton of cane ground.

Decreased sugar loss in filter mud is only one of the ways in which cane washing benefits. When the cane is muddy, the mud tends to polish the mill rollers, causing them to slip excessively. In a short time they become worn smooth and will not pull the cane through the opening between them. Instead they tend to slip against the cane, closing the passage for it, and thereby causing a "Choke". When this happens the mill must be stopped, the pressure on the rollers released, and the "Choke" cleared by hand. The time lost in removing "Chokes" is a loss to the factory, which is generally trying to operate at maximum throughput, both to economize on labor and operating cost and to get the crop in before a freeze occurs.

In addition, the mud removed by washing is kept out of the mill, thereby permitting more cane to be ground in its place. When rainy conditions are prevalent and the fields are muddy, increases in grinding rate of up to 35% have been obtained simply by washing the cane. This is attained with no additional equipment or labor and constitutes roughly an additional $6000 worth of product per day produced by increasing throughput (for the average mill).

In addition to a reduction in filter mud, decreased equipment wear, and increased grinding rate, the absence of mud in the bagasse produced by a mill is of value. Whether it results from better operation of furnaces burning bagasse, higher quality of chicken litter made from it, or better suitability for its use in making paper or wallboard, the better quality of bagasse produced from washed cane is a definite asset.

The average sugar mill surveyed used an average of about 1000 GPM of water for washing cane. The amount absorbed by the cane is small as compared to the total volume. Heretofore, mills other than those located on the Mississippi River or owning large areas of swamp land which can be used for disposal have been faced with the necessity of impounding all water used, or giving up the idea of washing cane. No success had been found in treating the effluent or minimizing its volume by re-cycle. Re-cycle was feared by the mills because it was thought that organisms would build up in the circulating water, which, if carried into the mill on the washed cane, would cause inversion of the sucrose in the raw juice. The material itself varies from coarse grit to fine collodial mud.

Thus, the problem was simply to either obtain about 3000 acre feet of disposal lagoon or not wash cane. Many factories were forced to stop operations during rainy periods because they were unable to dispose of the wash water.

The field survey showed, as its final result, that all effluents other than cane wash water could be successfully impounded or eliminated at the source.

CHAPTER V

THE DISPOSAL OF CANE WASH EFFLUENT

The difficulty in obtaining an adequate supply of harvest labor at a cost which the sugar industry could afford to pay was the principal factor motivating the development of the first cane cutting machine. Since no satisfactory method has been developed to clean cane mechanically as harvested, there followed the general practice of allowing the cut cane to remain in the field several days after which time the leaves and trash could be removed by burning. This procedure is not effective in rainy weather so that during such periods the grower must either deliver trashy cane to the mill or cease delivery until the cane is dry enough to burn. Because of the limited harvest season and the ever present freeze hazard deliveries are generally continued, and cane received by the mills during wet weather contains anywhere from 10% to 50% trash. With mechanization there has been an increase in the quantity of cane trash delivered to the mills and a general lowering of the quality of all work attendant on the harvesting operations[16]. This has resulted in a higher mud and trash content on the cane at all times, and particularly when it rains.

A study of the literature shows no work had been published on the disposal of cane wash effluent prior to this project. There are many works dealing with beet sugar waste treatment. A good summary of their problem is given by Southgate[21] and covers British, German and American beet factories. Although the beet process differs greatly from that for making cane sugar there are certain similar operations. The beets are washing prior to slicing and the effluent from this step is somewhat like cane wash water. A survey made in 1933[6] by the English pollution control authorities showed this effluent to be in the range of 3000 gallons per ton of beets, to have a B.O.D. of about 200 parts per million, and a population equivalent to about 50. A number of other references to beet sugar industry pollution are found in the literature. In an investigation by the Water Pollution Laboratory of England[2] it was found that at three factories at which transport water was being re-used the concentration of suspended solids was reduced by 92-98% by sedimentation. At other locations the removal was much less and varied with the location, apparently depending upon the nature of the soil. After sedimentation the liquid had an average B.O.D. of 700 to 2000 parts per million, much higher than for cane wash effluent. The liquid was far too polluted to be discharged to the British streams. Biological treatment was not used because it was believed to be too expensive. Generally this material has impounded in storage lagoons. Note that the water itself is re-cycled and only the settled sludge remains in the lagoons. No ill effects upon the sugar recovery are reported.

The general opinion of beet sugar investigators[10,12,20,23,29,3 is that the wash water should be re-cycled, that chemical clarification is not economically justified, and that lagooning is the practical method of disposal. Although bio-treatment is mentioned as a possibility, the land situation in the beet sugar areas is not so critical, and it has not been generally adopted in place of storage lagooning.

In general the work done on beet effluents is not applicable to cane sugar effluents because the concentrations in the beet effluents of B.O.D. are ten fold or more times that from the cane sugar factories. Also, the effluents from the process, other than condenser water and wash water, are not comparable. Although a large number of references concerning beet sugar wastes were investigated their usefulness is not great enough to give them further mention.

<u>Current Practices Survey</u>: It has been mentioned that the sugar factories of Louisiana were forced to initiate the practice of washing cane as it enters the mills. Although it was known that about half of the factories washed cane, no

reliable information was available as to the washing installations, periods of operation, quantities of water used, cost of the operations or benefits the operators felt were being derived. In order to clarify these points, a questionnaire was sent out to all mills in Louisiana inquiring what the various factories were doing.

Thirty-five answers were received to the questionnaire. Of these mills, 22 washed cane as a routine, 13 did not wash at all, and 2 of the 13 formerly had washed cane but were forced to stop because they had no disposal facilities. Other pertinent facts gleaned from the information received are summarized in Table III.

Based on the results shown in Table III, it became evident that the disposal of cane wash effluent was fast becoming the primary problem of the sugar industry in Louisiana. As the other effluents of pollutional nature had been evaluated and satisfactory ways had been devised to prevent them from being pollution problems any longer, it was decided to concentrate on the cane washing operation to make it more efficient to reduce the volume of effluent to be treated, and to devise the cheapest workable treatment method.

The chief disadvantage of washing cane for many mills lies in the difficulty of disposing of the effluent. If the

TABLE III

SUMMARY OF CANE WASHING PRACTICE IN LOUISIANA

Number of factories washing	22
Number of factories washing continuously	14
Average milling capacity; rated (tons/day)	2,360
Average milling capacity; actual (tons/day)	2,460
Average cane ground during season, 1955 (tons)	164,492
Number of days cane was washed	40
Source of water used for washing:	
Condenser effluent only	13
Fresh water only	4
Combination of both	5
Type of disposal facility:	
Mississippi River	4
Swamp	9
Impounding area	9
Depth when full (feet)	6.8
Number of days before full	1.3
Volume of water used for washing (GPM)	1,148
Location where cane washed:	
Feeder table (%)	22
Cane carrier (%)	48
Both places (%)	30
Mills washing cane every year (%)	86
Mills washing continuously (%)	64
Average water pressure applied to cane (PSIG)	42
Mills desiring improved washing installation (%)	96
Effects of washing on mill operation:	
Reducing number of "chokes" (%)	81
Improved bagasse blanket (%)	70
Improved juice clarity (%)	88
Reduced filter press mud volume (%)	90
Reduction in filtrate re-cycle problems (%)	83
Loss of obtainable sugar by washing (% No)	73
Yearling Washing expense ($)	1,850
Reduction in mud (pounds per ton cane)	47
Effluent purified by treatment	None

Year cane washing was initiated:
1941 (1); 1945 (1); 1946 (2); 1947 (5); 1948 (3);
1949 (1); 1950 (1); 1952 (1); 1953 (2); 1955 (1).

(These are answers received to a questionnaire sent to all Louisiana sugar factories at the end of the 1956 season) effluent may be economically disposed of without treatment or impounding costs then washing is a certain profit maker. If, on the other hand, treatment is necessary the decision to wash or not to wash rests on an economic balance. Usually, when not justified by sugar saving alone, washing will be necessary in order to keep the mill in operation during rainy seasons.

Cane may be washed in either of two locations: (1) on the feeder table, or (2) in the cane carrier. Some mills advocate washing on the table in order to let the maximum amount of water drain off of the cane before it enters the knives. Others, as a matter of convenience, wash on the carrier itself. There is no generally proven best location for the washing station. Many mills, in fact, wash the cane twice, once on the apron and again after it falls onto the carrier. It is felt by many that the bundle is turned over and redistributed when falling onto the carrier in such a manner that a new section of the bundle is exposed to the sprays.

Cane wash effluent is disposed of in one of three ways: (1) pumping it into a large river such as the Mississippi, where it will be quickly diluted; (2) allowing it to flow onto uninhabited swamp lands where it can decompose without becoming a nuisance or health hazard; or (3) impounding it in lagoons until biological stabilization has removed all pollutional material. Once the effluent has purified itself it may be emptied into a stream.

Unfortunately, only those mills along very large rivers, such as the Mississippi, can dispose of their effluent by method (1). In many cases the cost of erecting pumping installations to pump the material over the levee is made prohibitive because of strict government specifications set up by the U.S. Corps of Engineers. For this reason many mills find it more economical to use method (2) even though they are located next to a river. Method (2) is the cheapest method of effluent disposal, but the location of some mills is such that prohibitively long ditches and lines must be laid to carry away the effluent. Impounding in a lagoon, (3), is the most expensive method. In addition to piping and pumps, extensive earth works must be erected. Over a period of years the pond will fill up with sediment. Heavy equipment must be employed to remove the silt from the bottom during the summer time when the pond is not in use. Many mills are situated in areas where real estate development in recent years has used up all available land. Several mills once considered isolated are now in the center of communities.

People in these communities frequently are employed in oil production, supply, or other jobs not related to sugar manufacture. Consequently, complaints are not suppressed when the mill causes annoyance to the village from odor, dust or smoke.

In addition to the fact that construction of impounding lagoons is costly, many mills are unable to buy additional acreage adjacent to the factory site at any price. Because of the inability to build new lagoons or enlarge existing ones it has become necessary to devise ways to reduce effluent volumes and also to devise ways of utilizing the lagoons in purifying wastes.

Volume Reduction: Generally, cane washing is carried out with little concern given to the volume of water used to wash the cane. Frequently the sprays are operated when no cane is actually being washed. This causes a needlessly high volume of effluent.

Most mills have cut down this waste of clean water by installing quick-closing valves which permit the operator to stop the flow of spray water when no cane is being washed.

There are three general methods of reducing the volume of effluent cane wash water. They are (1) continuous re-cycle of wash water; (2) continuous re-cycle of wash water with intermediate settling; and (3) multi-stage washing. All of these methods involve some form of re-use of the effluent from the washing operation.

Total re-cycle involves merely pumping the wash water back over the cane without any other treatment. It is the simplest and cheapest procedure, but its short coming is an excessive build up of solids in the circulating system with accompanying undue wear on piping and pumps, loss of dirt removal ability, and possible contamination of the juice squeezed from the freshly-washed cane. Although the water could be re-cycled for several hours before bacterial action became excessive there is a great concern that it might cause serious trouble within the plant. In the past many factories not observing strict cleanliness around the mills have found that growths of certain organisms caused the loss of sucrose in the raw juice through inversion. Such changes result in a loss of sugar production. For this reason no factory was willing to permit experimentation using total re-cycle of their wash water.

Observations made of the once-through washing carried on by many of the mills indicated that re-cycle, if accompanied by primary type treatment, might prove practical provided the system is purged every few hours to prevent bacterial development. To try out this idea the loan of a

6' x 8' "Graverette" pilot plant clarifier from the Graver Tank and Manufacturing Company was arranged. The unit was installed at the Sterling factory located in Franklin, Louisiana. The unit could not be made to operate on cane wash effluent because of the heavy mud loading obtained from the wash water. The sludge draw-off would not accomodate the mud as fast as it was collected. In order to improve this condition a grit settling tank was installed ahead of the clarifier. The factory washed cane only when necessary and the unit could not be operated continuously.

Lime, Alum, and various settling aids were used in an effort to clarify the wash water. It was found that a strong dosage with lime only provided as good a treatment as lime in combination with other chemicals. The high chemical dosage required (500 ppm lime) made it un-economical for clarifying the cane wash effluent.

In conjunction with the pilot plant clarifier operation a large number of jar settling tests were made using various settling aids. This was done to determine preliminary dosages to be added to the pilot plant. Samples of wash water were taken from other nearby factories for evaluation. The results of these settling tests, will be found in the appendix.

Solids determinations were run on a number of cane wash effluent samples. Table IV contains the solids analysis of 23 sets of samples. In addition to solids, the sugar content of each sample was estimated using the alpha-naphthol test as an indicator. At high concentrations it was possible to obtain direct sugar content readings by concentrating the material under vacuum, making a direct polarization on the concentrated sample with a saccharimeter. The solids pick-up, although subject to considerable variation, tends to average about one-half per cent. The sugar content of the water is generally in excess of 200 ppm. There seems to be no relation between added solids pick-up and the solids content of the entering water. This makes re-cycle more attractive because it shows that dirty water should wash about as well as fresh water under the conditions existing. Evidently the material tested had a solids content well below the limiting value where the water could take up no more.

Cane Wash Water Re-Cycle: Multi-stage washing consists of spraying fresh water on the cane as it enters the factory. The effluent from this operation serves as make up water for a preceding set of sprays which spray the dirty cane at a location further down the conveyor from the factory. The effluent from this outer set of sprays is discarded from the washing system in an amount equal to the influent to the fresh

TABLE IV

CANE WASH WATER SOLIDS DATA, 1955

Date	Solids, %			Centrifugal	Sugar (ppm)	
	Supply, %	Wash, %	Pickup, %	Solids, %	Supply	Wash
			Sterling Sugars, Inc.			
Nov. 29		0.6238 S			56	183
Dec. 2		0.9637 S			220	356
8		0.7036 S			63	171
17	0.0351 T	0.7831 T	0.7480		20 AN	200 AN
18	0.0182 T	0.4210 T	0.4038		50 AN	200 AN
19	0.0388 T	0.5838 T	0.5450		100 AN	200 AN
20	0.0291 T	0.7398 T	0.7107	0.5	50 AN	200 AN
21	0.0207 T	0.6207 T	0.6000	0.3	15 AN	200 AN
22	0.0359 T	0.5846 T	0.5487	1.35	20 AN	200 AN
			St. Mary Co-operative			
Dec. 9		0.7326 S			56	167
12		1.0360 S			78	548
13	0.0291 T	0.8437 T	0.8146		46	248
17	0.0027 T	0.3273 T	0.3246		none	150 AN
18	0.0027 T	0.1253 T	0.1226		20 AN	200 AN
19	0.0287 T	0.5329 T	0.5042		20 AN	200 AN
20	0.0035 T	0.2581 T	0.2546	0.4	50 AN	200 AN
21	0.0113 T	0.5324 T	0.5211	0.4	15 AN	200 AN

(Continued on next page)

TABLE IV

(Continued)

Date	Solids, % Supply, %	Wash, %	Pickup, %	Centrifugal Solids, %	Sugar (ppm) Supply	Wash
			Iberia Sugar Co-operative			
Dec. 7		0.2020 S		0.75 set.	76	356
13	0.0329 T	0.9370 T	0.9041		193	306
17	0.0047 T	0.8317 T	0.8270		20 AN	200 AN
18	0.0412 T	0.9342 T	0.8930		50 AN	200 AN
19	0.0367 T	2.0797 T	2.0430		200 AN	1.04% DP
20	0.0401 T	0.3890 T	0.3489	0.6	100 AN	200 AN

S = Suspended Solids AN = Sugar estimated by Alpha Naphthol

T = Total Solids set. = Solids settled out in 45 minutes

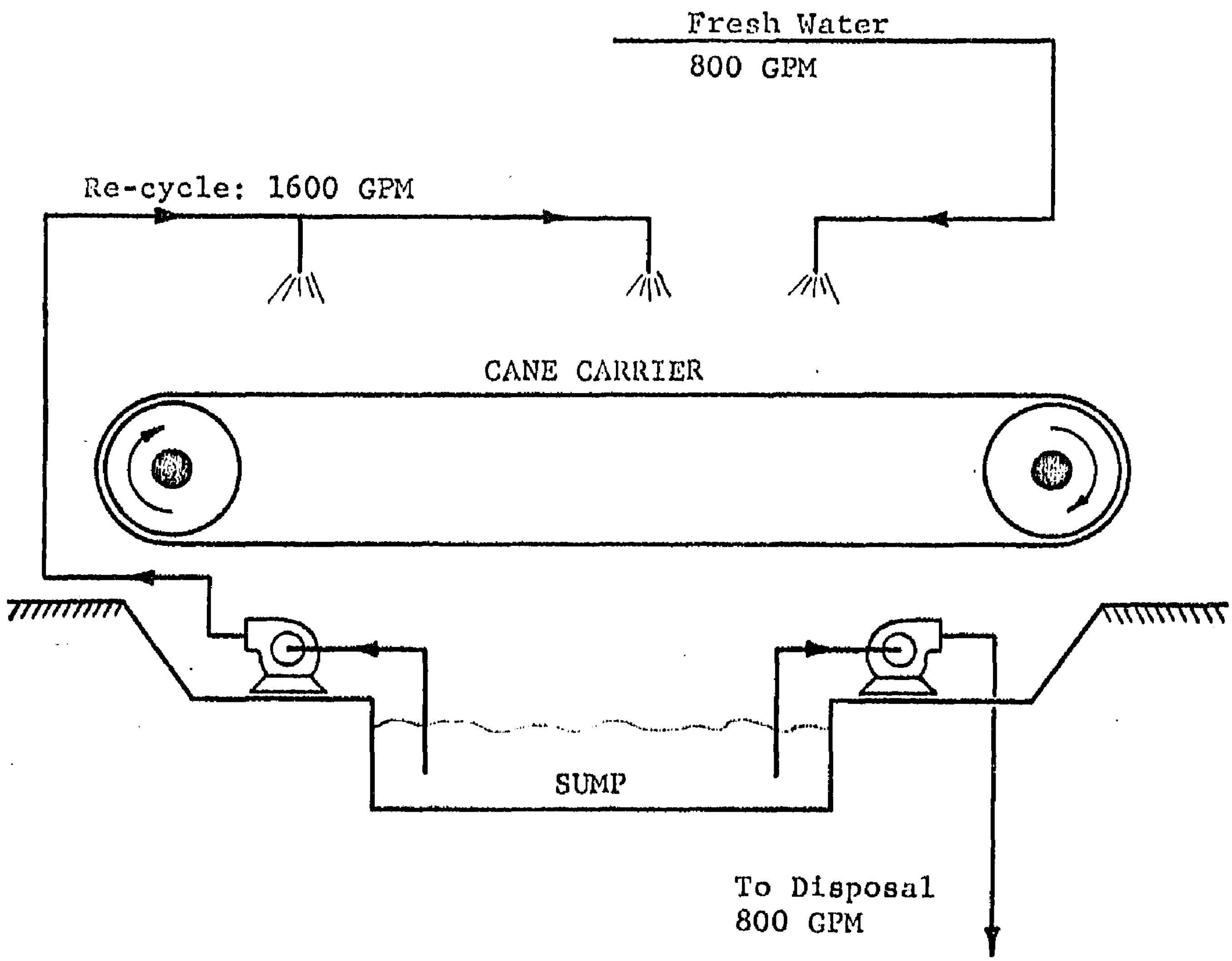
Centrifugal = Solids centrifuged out in 5 seconds at 240 g's water sprays. In this manner the cane is washed first by used water to remove most of the dirt and trash and is then given a clean rinse before it enters the factory. This represents a stepwise approach to the ideal concept of countercurrent operation.

Prior to the 1957 season, Caldwell Sugars Co-operative, Inc. installed such a system. About 800 GPM of fresh water was sprayed over the cane at a location a short distance ahead of the revolving cane knives. A second set of sprays circulated water at a rate of about 2000 GPM over the cane entering the wash rack. This installation is shown diagrammatically in Figure 9.

A number of samples were taken from various locations in the system and analyzed for solids, B.O.D. and O.C. The average values obtained from these analyses are shown on the diagram in Figure 9. A total of 2800 GPM of water was applied to the cane and yet the effluent volume was only 800 GPM. No ill effects were observed in the operation of the sugar factory. Another advantage obtained is the more concentrated effluent. Since the rate of bio-stabilization is a function of the concentration of putrescible material in solution, the overall rate of treatment is aided by having a concentrated feed material, thereby providing more economical treatment of

FIGURE 9

DIAGRAM OF MULTI-STAGE CANE WASH INSTALLATION
AT
CALDWELL SUGAR COOPERATIVE



the wash effluent.

Physical Treatment: The pre-disposal treatment of cane wash water is of two types, physical and chemical. By physical methods, such as screening, settling or filtering, it is possible to remove a high fraction of the total ultimate B.O.D. This is necessary not only to remove any solids which tend to fill up later treatment facilities, but because, if not removed, the cellulose or woody material in the cane wash water slowly decomposes with accompanying liquification, thereby re-polluting the waters. A layer of this material deposited on the bottom of an impounding lagoon will exert a continual B.O.D. as it slowly decomposes over a period of many months and thereby prevent the water from reaching a low enough B.O.D. concentration to be released to a public stream. It was shown in Table II that approximately 60% of the total solids content of cane wash effluent could be removed by settling. Therefore physical treatment is a necessity for economic and efficient effluent treatment.

Simple Settling: Settling is necessary as a first step in any treatment. This operation serves to remove grit and sand from the water prior to any treatment or re-use. Experience has shown that the material that can be removed from cane wash effluent by settling will be eliminated quite rapidly. The solid material not removed by settling is generally of a colloidal nature and requires some form of chemical treatment to permit its removal. Chemical clarification does not appear to be economic under present volume-benefit-cost conditions.

Screening: Another treatment required regardless of the future use of the wash water is screening. Generally the effluent is passed through a screen having 1/4 inch openings to remove the large amounts of woody material which is washed from the cane. This is done to prevent clogging of pumps, valves, and pipes. Also, if it is not removed, the woody material will deposit on the bottom of any treatment or storage pond and slowly decompose to exert a continuous pollutional effect upon any water contained therein.

Treatment, Chemical: Chemical methods of waste treatment consist of chemical clarification, chemical oxidation, and biological oxidation.

Clarification: Clarification is often confused with the operation of simple settling. The difference lies in the chemical floc utilized in a clarifier to filter and absorb material contained in the liquid passing through. Colloidal materials may be removed by clarification whereas they cannot be removed by simple settling. From the work done at the

Sterling factory, it was discovered that the coarse material contained in cane wash effluent could be readily settled but it was exceedingly difficult to effect clarification of the liquid. It was found that various settling aids gave no greater benefit than the use of lime alone. The cost of the amount of lime needed for clarification rendered the operation un-economical.

Chemical Oxidation: Chemical treatment has been reported involving the use of chlorine and ozone in the treatment of factory effluent from beet mills. It was found that the cost of constructing an ozone plant of adequate size to handle the average cane mill effluent was over $250,000 and that the cost of producing the amount of ozone needed to treat cane wash water alone was about $1300 per day. These figures were obtained from representatives of a leading ozone concern. It was rather disappointing to find out that the cost of the ozone units were as great as the value of some of the sugar mills.

The use of chlorine was discussed previously. Its use is un-economical even from the ideal concept of using it to chemically oxidize the organic material in the effluent. Experience with chlorinated waters has shown that certain organic chlorine compounds may be formed which impart a very medicinal taste to the water, rendering it non-potable. The high chloride concentration resulting could be injurious to aquatic life in the receiving streams.

Bio-Oxidation: The only method remaining that might have use in treating the wash effluent is that of bio-stabilization. This is "nature's own method" for disposing of organic wastes and basically involves allowing micro-organisms to act upon the waste before it is discharged and thereby confining the pollutional effects to a small, controlled area. Cost considerations and the slim profit margin found in cane sugar manufacture dictate that only the minimum treatment is to be considered. Generally proven processes as activated sludge and trickling filters are out of reach economically. Therefore, the treatment of these materials must be done using only earthen pits and ponds, a few pumps and possibly a spray for aeration.

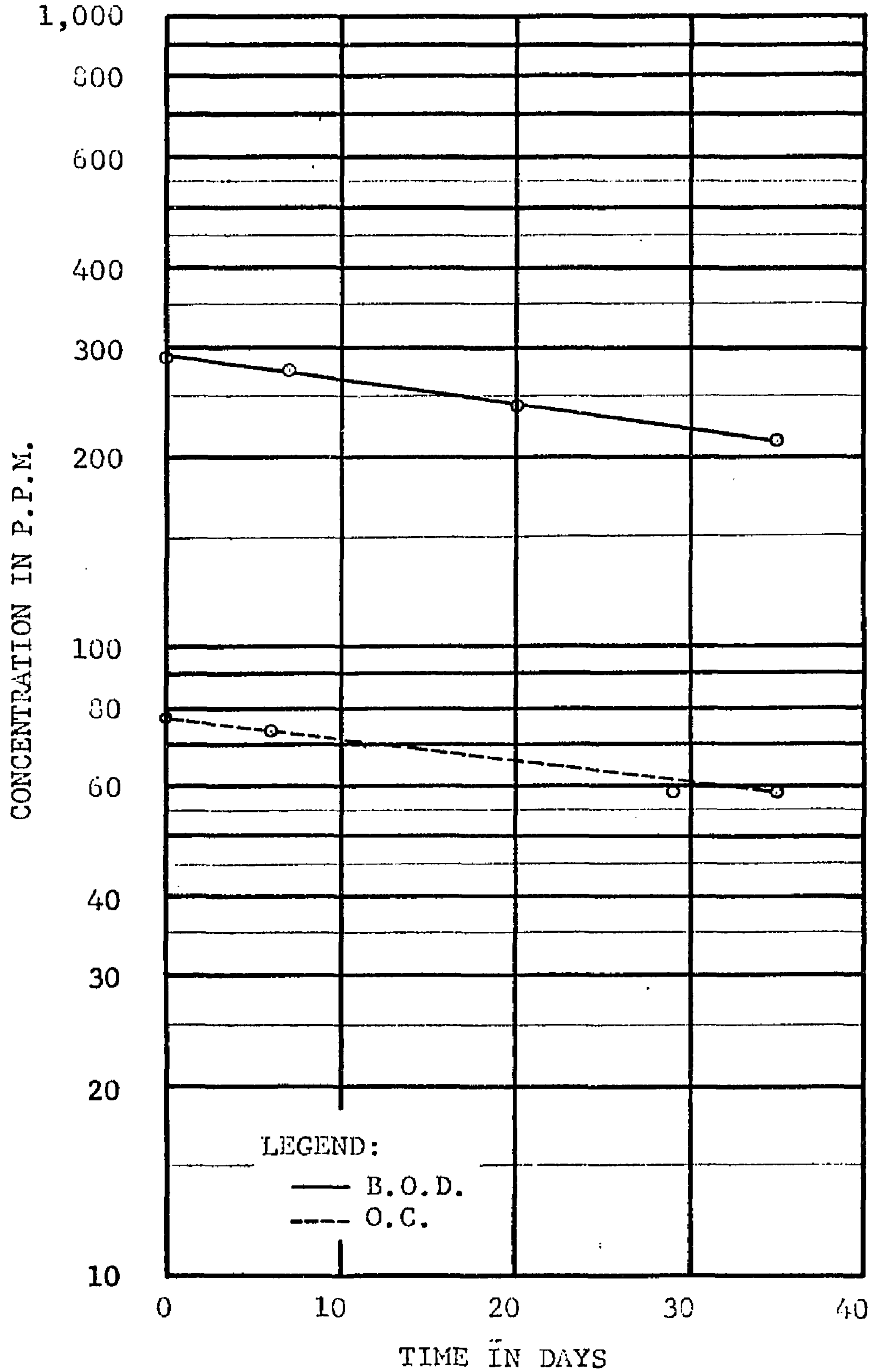
At the start of the program it was evident that the existing treatment and/or impounding ponds were woefully inadequate. The pond depths ranged from six to fifteen feet, much too great for aerobic decomposition. Solids were not removed from the wash water before it was impounded. These solids deposited on the bottom in a thick layer of decomposing sludge, its solid material slowly dissolving through anaerobic decomposition to replenish the B.O.D. of the water above. No effort was made to supply aeration nor to provide the proper nutrients for biological stabilization to occur at its optimum rate. Since there is a steady replenishment of the dissolved pollutional material, the effluent will but slowly reach a quality such that will permit its release to a receiving body of water without bad effect. Figure 10 illustrates this point. It is a plot of B.O.D. against time as observed in the Iberia impounding lagoon where solids have been allowed to enter with the cane wash effluent. Ordinarily a steeper line would result. However, the continued release of pollutional material from the solids deposited on the bottom serves to maintain the B.O.D. at a high level. No fresh effluent was being added during the period covered by this data. The action in the pond is anaerobic because of the inability of oxygen to diffuse through the surface rapidly enough to satisfy the B.O.D.

Because of the previously stated operational and economic limitations it is believed that the treatment of the sugar mill effluents must be made in such ponds and at a minimum outlay for equipment and operation. To do this it is necessary to study the factors affecting optimum biological stabilization to minimize the size of installation needed, allow rapid disposal, and eliminate odors.

FIGURE 10

IBERIA IMPOUNDING POND

12/30/57 to 2/3/58



CHAPTER VI

BIO-STABILIZATION STUDIES

The Need for a New Process

The cheapest method of treatment for cane wash effluent is the use of a properly designed, properly operated oxidation pond. A trickling filter or activated sludge treatment plant has a fixed investment cost of at least $150,000. This high cost combined with the need for experienced operating personnel, prices it out of the seasonal, low profit raw cane sugar industry in Louisiana.

Often mills must wash cane in order to operate. To get the crop in they must be able to dispose of the effluent in some type of pond. The available ponding areas are very limited and by order of the Stream Control Commission a factory must not discharge more than 1.5 pounds of B.O.D. per ton of cane ground[31]. This leads to the conclusion: the mills need to develop a cheap process for treating their cane wash effluent by oxidation ponding. Otherwise, they may go out of business and with them the livelihood of thousands of the inhabitants of South Louisiana.

Functions of Micro-organisms in Treatment

The key to this waste treatment problem is a study of the factors affecting the functioning of the organisms which perform the so-called bio-stabilization. There is a wide range of types of micro-organisms, some having a high rate of metabolic activity. The common types are: unicellular yeasts, protozoa, fungi and bacteria. Bacteria have the highest rates of metabolism among micro-organisms and therefore are of great importance in waste treatment.

Different bacteria utilize different methods of attack on the same particle of waste. This gives rise to the varied products of bacterial activity and to the apparently involved and complicated metabolism of the order as a whole.

When an organism encounters a material which it can utilize as food it begins to grow in size until eventually division takes place with the formation of two cells from one. The rate at which division takes place depends to a large extent upon the nature of the medium. An organism such as the common Escherichia Coli (found in human intestines) living in a rich medium can divide once every 10 to 15 minutes. One single organism can give rise to over one million progeny in five hours. This high rate of synthesis takes place at the expense of the environment which must supply all of the raw materials. The material used as food may not always be present in readily useable form so the organism must break down the compounds present to forms that may be assimilated.

The synthesis of the chemical complex of the bacterial cell involves the expenditure of energy. This is obtained by the degradation of energy-rich substances in the environment. The end products of this degradation are carbon dioxide and water. If insufficient dissolved oxygen is provided the reaction is much slower, and fermentation is an intermediate step. Although the end products will eventually be the same as for aerobic oxidation, volatile acids, methane, and hydrogen sulfide will be formed, creating an odor nuisance.

Bacteria have a wide distribution in nature. The only places free from them are those where a sterilizing influence is at work. It is not necessary to search for the types needed to act upon the sugar mill effluents. They are already present around the factory, in the ponds, and on the cane shipped to the mills.

Mechanism of Biological Stabilization

There are three types of reactions associated with these biologically active slimes and sludges. These are oxidation, cell synthesis, and respiration.

Straight oxidation provides the necessary energy for life processes. This can be conveniently represented by the equation[7]:

$$C_xH_yO_z + (x + 1/4y - 1/2z)\ O_2 \rightarrow xCO_2 + 1/2y\ H_2O + \text{heat}$$

Additional cells are synthesized, according to an equation of this general form:

$$n(C_xH_yO_z) + nNH_3 + n(x + 1/4y - 1/2z - 5)\ O_2 \rightarrow (C_5H_7NO_2)n + n(x-5)\ CO_2 + 1/2n(y-4)\ H_2O \pm \text{heat}$$

Microbial sludge produced undergoes continuous auto-oxidation (endogenous respiration) like this:

$$(C_5H_7NO_2)_n + 5nO_2 \rightarrow 5nCO_2 + 2nH_2O + nNH_3 + \text{heat}$$

The empirical formula for cellular material ($C_5H_7NO_2$) is representative of the ratios of the primary constituents found in biological sludge[14].

Biological floc is a collection of micro-organisms, such as bacteria, yeasts, molds, algae, protozoa, rotifiers, worms and insect larvae, in a gelatinous matrix.

Stages of Biological Growth

In the presence of oxidizable organic matter, bacterial growth undergoes several stages. First is a "lag" or induction period. This phase is omitted where there is a continuous flow of material into a growth that has been previously formed.

Another early phase is the storage period during which B.O.D. is removed from solution but not immediately metabolized. As time goes on and the soluble B.O.D. in the waste is removed by the bacterial growth the micro-organisms consume the stored material for metabolism and growth.

The next phase is the "log growth phase" and is the period during which regular and maximum multiplication of cells is taking place. Growth occurs by the division of one cell into two cells. Its rate is limited by the concentration of food present at any particular time. With ample food the growth is logarithmic, because at any given time the rate of growth is a function of the number of cells present.

Next is a deceleration phase in which division of cells occurs at slower intervals. It has been shown[9] that the rate of cell growth below the maximum rate is dependent upon the B.O.D. concentration and hence the rate of utilization or stabilization is a function of B.O.D. concentration. Therefore a mean rate constant, representative of the over-all process during this phase, may be related to the food supply or B.O.D. This is the basis of the first order reaction velocity relations common to biological decomposition.

In the final stage there is little B.O.D. removal from the solution. The biological growth is decomposing at a rate greater than synthesis. Endogeneous respiration is said to be taking place.

In most industrial operations the material is kept in a growth phase in order to effect maximum B.O.D. removal. The excess growth is settled out and discarded and the rest is used to seed the incoming effluent. This is the basic operation carried out in the so-called activated sludge operation. For this work it is necessary to effect a similar action with only the help of earthen settling basins.

Preliminary Treatment Studies

In order to develop a cheap workable process for treating the cane wash effluent it was necessary to study the bacterial decomposition of this material to find ways to optimize the action taking place. There are a number of factors which limit the biological stabilization rate. The most important of these are aeration, temperature, nutrient addition, prior solids removal and the B.O.D. concentration in the feed. Laboratory studies were undertaken in order to evaluate these effects upon the stabilization of cane wash effluent.

The first of these studies was made using an open beaker filled with a mixture of cane juice and cane wash water. The initial mixture had an O.C. of 410 ppm. There was no temperature control and no aeration other than that received by surface absorption from the air. The temperature ranged from

27° to 32°C. A small amount of "seed" consisting of some old cane wash water was added to provide the micro-organisms found around cane sugar factories. The results of the 160 hour run are shown graphically on Figure 11, a chronological plot of the logarithm of the fraction of O.C. remaining at the time each analysis was made. When expressed this way the slope of the first order reaction is proportional to the reaction rate constant at a given time.

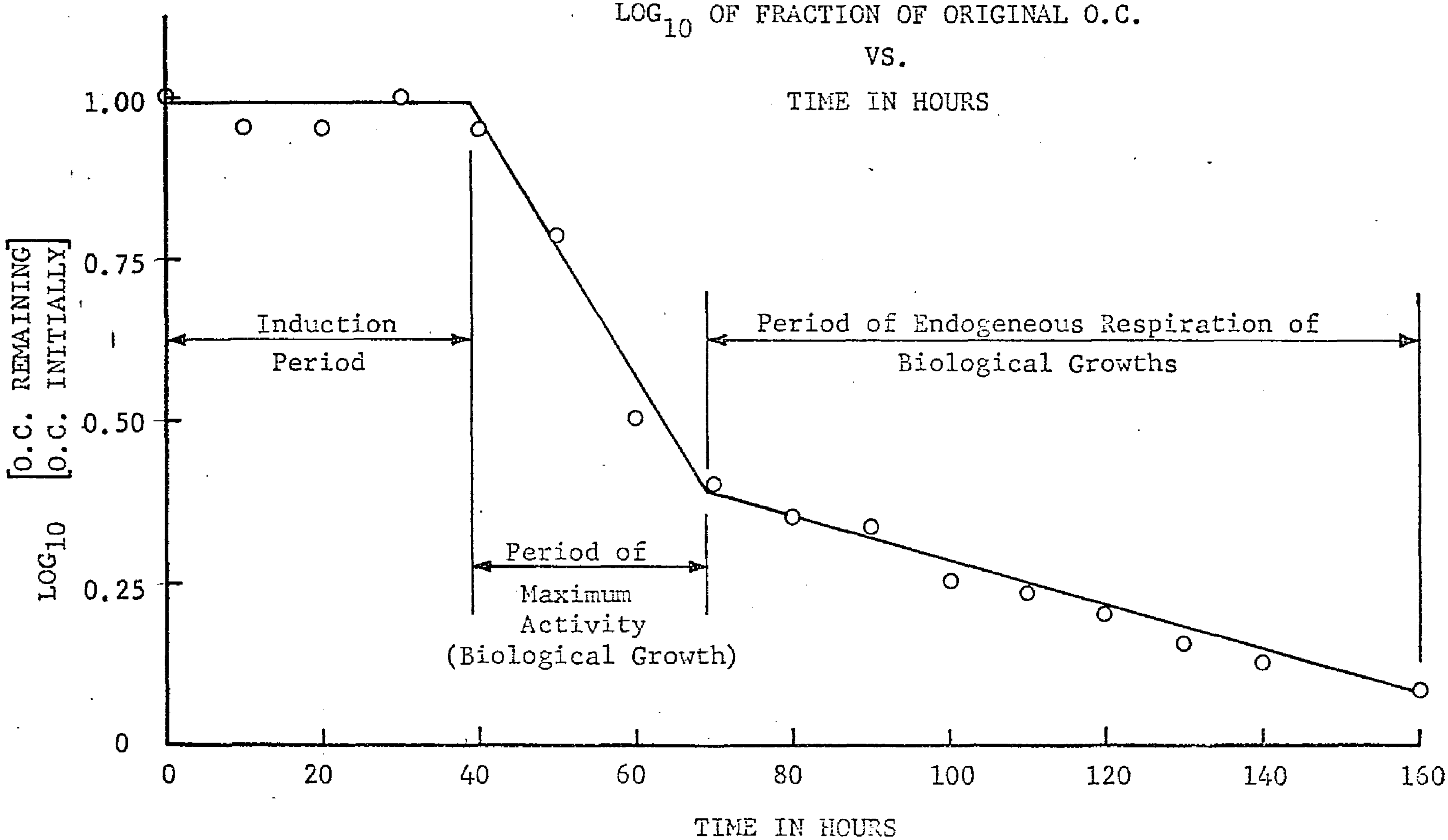
There were three rather distinct steps. The first was the incubation period lasting about 30 hours. During this time the bacteria were becoming "acclimated" to the feed material and those best suited to metabolize it formed their colonies and superceded others. The second step is that during which the maximum metabolism took place. During this time the action was first order and showed a rate constant of .46 hours^{-1}. After about 30 hours the O.C. had dropped to 100 ppm and the rate constant assumed a value of .08. This remained constant for the rest of the run.

This initial study, although not very precise, showed that two steps existed, each having its own rate constant. It was felt that further study was warranted to find ways to keep the operation at its maximum rate by evaluating those factors affecting the rate and thereby optimize the treatment

FIGURE 11

FIRST STABILIZATION TEST

LOG_{10} OF FRACTION OF ORIGINAL O.C.
VS.
TIME IN HOURS



process.

A second trial was made using nine reactors. Feed material was similar to the first. Four of the reactors were adjusted daily to a pH of 5, 6, 7, or 8, respectively, using ammonia. Four others were adjusted to corresponding pH's using lime. The remaining sample received no additives and served as a blank. All nine were placed in open beakers.

The results of this series are shown in Table V. No clear-cut conclusions as to the optimum pH of nutrient can be made from this run because other conditions, mainly temperature, were not controlled.

Supply of Raw Material

During the spring of the year when the sugar mills were closed a series of trials was undertaken to obtain kinetic data on the stabilization of actual sugar mill effluent. In order to have a ready supply of raw material arrangements were made with the Audubon Sugar Factory to place a number of five gallon cans of effluent in frozen storage. About 125 gallons of cane wash effluent from the various factories was frozen and kept at -20°F until needed. Since the studies were aimed at dissolved B.O.D. removal the effluent was filtered through coarse filters to remove settleable solids just prior to use.

TABLE V

EFFLUENT STABILIZATION STUDY - EFFECT OF pH ON STABILIZATION RATE
SYNTHETIC EFFLUENT

Hours	0	13.5	36.5	61.5	85.5	109.5	142.5	167	186	234.5	258.5	282.5	332	Average pH
N-8														
pH	6.6	6.8	4.3	7.1	7.6	7.7	7.7	7.9	7.8	7.7	7.8	8.0		7.25
O.C.	410	376	345	75	68		64		35				26	
BOD	369				375									
N-7														
pH	6.6	6.5	4.2	6.7	7.3	7.3	7.3	7.5	7.5	7.7	7.7	7.8		7.03
O.C.	410	378	313	120	110		33		31				15	
BOD	369				345									
N-6														
pH	6.6	5.7	4.3	5.1	6.6	6.8	6.8	7.1	7.3	7.3	7.4	7.5		6.54
O.C.	410	378	400	60	68		42		27				14	
BOD	369				479									
N-5														
pH	6.6	6.0	4.6	4.3	5.8	6.4	6.0	6.1	5.9	7.0	7.1	0.0		5.98
O.C.	410	378	405	55	51		42		31				0.0	
BOD	369				345									
Blank														
pH	6.6	0.0	4.5	4.3	4.2	4.4	4.1	4.3	4.3	4.4	4.4	4.6		4.55
O.C.	410	378	390	220	81				44				36	
BOD	369				544									

(Continued on next page)

TABLE V
(Continued)

Hours	0	13.5	36.5	61.5	85.5	109.5	142.5	167	186	234.5	258.5	282.5	332	Average pH
C-8														
pH	6.6	6.3	4.9	5.3	6.8	8.6	8.0	7.8	8.0	7.6	7.9	8.2		7.17
O.C.	410	371	427		68		56		29				17	
BOD	369				547									
C-7														
pH	6.6	6.1	4.8	5.3	6.4	7.3	7.2	7.2	7.3	7.5	7.5	7.7		6.74
O.C.	410	371	405	105	59		37		135				23	
BOD	369				564									
C-6														
pH	6.6	5.8	4.5	4.9	5.5	6.6	6.8	6.9	6.9	7.2	7.4	7.7		6.40
O.C.	410	378	393	135	81		69		21				55	
BOD	369				544									
C-5														
pH	6.6	6.1	4.5	4.6	4.6	5.3	5.1	6.7	7.0	7.1	7.3	7.7		6.05
O.C.	410	378	400	60	81		40		2				126	
BOD	369				524									

N = Samples whose pH was adjusted to ind. pH value with NH_4OH
C = Samples whose pH was adjusted to ind. pH value with $Ca(OH)_2$

Seeding: Small amounts of liquid from the previous run were added to supplement the population of natural micro-organisms by "seeding". Although this was not strictly necessary it was a precautionary measure insuring the presence of the micro-organisms acclimated to this type of material and thereby shortening the "lag" period during which the necessary growths form.

Flow Reactors: It was decided, as a result of the two preliminary trials, that a flow type reactor might be a better approach to the problem. Once equilibrium was reached only one set of analyses would be required to evaluate the operation at any given set of operating conditions.

Initially, it was hoped to operate several of the flow reactors in series and thereby simulate the actual holdups found at certain plants. In this manner the optimum pond holdup or "space velocity" would be determined. The first reactor consisted of three 4000 ml. glass beakers connected by inverted U-tubes. Feed was supplied from an elevated 5-gallon glass jar by means of a syphon. This jar was packed in ice to prevent decomposition of the feed. The rate of feed addition was adjusted by means of a stopcock. The level in each reactor could be set by adjusting its height relative to the draw-off and thereby change its holdup volume. The temperature of the reactors was controlled by a thermostat connected to a room air-conditioner. Several trials were made to "bracket" the range of flows and B.O.D. involved. Unfortunately it was not possible to maintain a constant flow because of plugging of the stopcock by sediment or slimes. This set-up was dismantled in favor of an improved type of pilot plant shown in Figure 12.

In this arrangement an adjustable rate pipetting machine was used as a low flow rate, precision pump. The reactors were immersed in a $\pm$ 0.01°F "Sargent" constant temperature water bath. The draw-off arrangement was similar to that used in the first unit. Cooling was supplied to the bath by water circulating through coils attached to the freezing compartment of a household refrigerator. Feed was stored in a glass jug packed in ice contained in a large "picnic cooler".

Several "dry runs" were made using domestic water to check the temperature and flow regulation. This was satisfactory and so cane wash effluent was charged to the unit.

A total of three sets of runs were made. Table VI shows data obtained from run #2 of the three. Note that in this arrangement the effluent from one reactor was fed to the following one. This had the advantage of allowing determinations at three concentrations to be made at one time. Space velocity

FIGURE 12

SCHEMATIC DIAGRAM OF FLOW REACTORS
SHOWING THERMOSTATIC WATER BATH, REFRIGERATION,
AND ADJUSTABLE RATE FEED PUMP

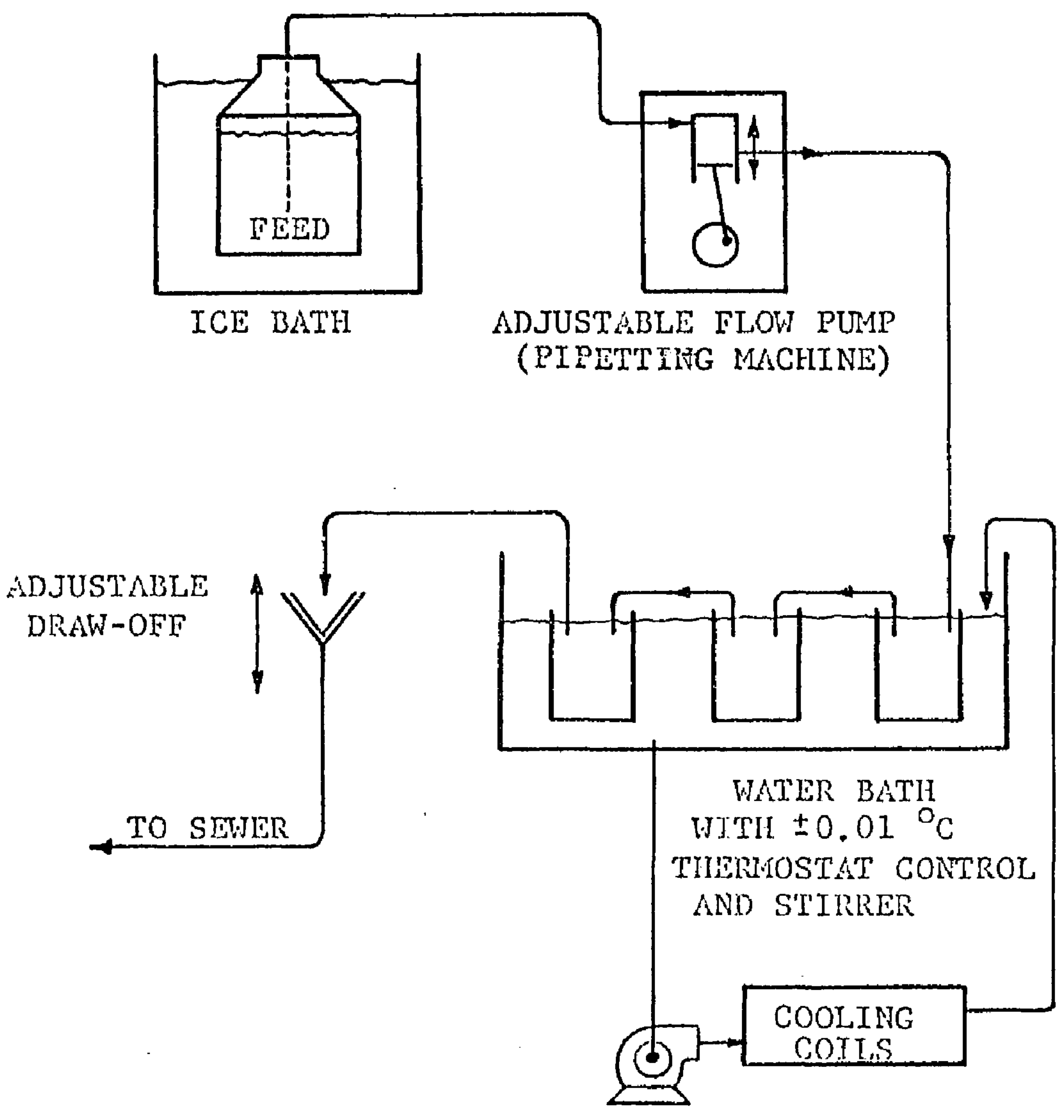


TABLE VI

EFFLUENT STABILIZATION PILOT PLANT

Data: Run No. 2

Reactor Volumes: 1500 cc. each

Total Hours	Temperature °C	Flow Rate gms./min.	O.C., ppm			
			Feed	Reactors		
				1	2	3
0	24.0	2.0	70	60	60	60
77	24.0	2.0	66	51	60	
120	22.3	1.9		87	75	80
144	22.1	2.1	85	75	69	64
168	26.1	2.1	77	68	59	61
264	23.5	1.9	73	55	48	46
288	24.0	2.1	74	54	51	48

Rate constants based on above data:

K_1 0.35 hr^{-1}

K_2 0.02 hr^{-1}

K_3 0.02 hr^{-1}

could be adjusted by the depth setting for each reactor.

The arrangement proved to be un-workable for several reasons: it was difficult to maintain continuous flow due to plugging of the tubing and the formation of gas bubbles in the feed line which "vapor locked" the reciprocating check-valve type pump. The refrigerator proved itself incapable of maintaining the temperature at the set point as is evidenced on the data sheet. Finally the refrigerator burned out and it was therefore decided to abandon this setup in favor of one more dependable and more easily controlled.

Non-flow Reactors

Because of the difficulties with the constant flow feeding arrangement it was decided to give up the flow reactors and go back to batch studies. This method was admittedly just as good but required the taking of a great number of samples to adequately determine the stabilization curve needed to calculate a reaction velocity constant.

In order to supply adequate cooling, the apparatus was relocated to a point next to a 3-ton water cooling unit used for student laboratory experiments. This unit supplied more than enough cold water to maintain the desired temperature in the water bath.

Three batch reactors were immersed in the constant temperature bath. One was sealed, another was open to the atmosphere, and the third was aerated by compressed air supplied below the surface through a porous diffusing stone. Three sets of runs were made. Two of these were negated when the coolant valve drifted shut during the night and the thermostat stuck at the same time, thereby pasteurizing the bacteria. The data from this run are tabulated in Table VII. Note that the aerobic reaction showed a much faster action than either the anaerobic or the open one. Note also that the B.O.D. of the sealed reactor was only slightly reduced at the end of the run whereas the other two had B.O.D. values of about 10% of the original feed. The feed for all of these runs consisted of filtered cane wash water which was collected during the grinding season and kept frozen at -30°F until needed. Although the O.C. of the sealed material dropped 340 ppm, the B.O.D. dropped only 29 ppm. Because of this it was decided to run both types of analyses (B.O.D. and O.C.) on the succeeding runs.

The thermostatic bath which had proven un-reliable after seven attempted runs was discarded. In its place a B.O.D. incubator having a $\pm$ 0.1°C thermostatic controller was obtained for use as a constant-temperature housing for the reactors. Filtered compressed air piped into the incubator

TABLE VII

EARLY PILOT PLANT RUN NO. 3

Feed: Frozen Cane Wash Water, Filtered

Total Hours	Date	Starting Time	I Open		II Aerated		III Sealed	
			B.O.D.	O.C.	B.O.D.	O.C.	B.O.D.	O.C.
0	Nov. 22	11 AM	509	470	509	470	509	470
28	23	3 PM		542		542		542
75	25	2 PM		334		431		389
57	24	8 PM		425		525		490
825	25	9:30 PM		332		396		348
95	26	10 AM		371		310		334
100	26	3 PM		277		279		320
109	26	12 PM		197		191		301
178	29	9 PM		231		172		249
200	30	7 PM		235		237		256
243	Dec. 2	2 PM	176	191	146	168	155	207
263	3	10 AM	138	191	168	243	165	220
292	4	3 PM		115		142		172
311	5	10 AM		122		112		143
413	9	4 PM		94		111		164
435	10	2 PM		91		69		133
469	Jan. 6	2 PM	75		38			
494	13	3 PM	50	95	55	86	480	130

for aeration purposes was supplied to the reactors through individual diffuser stones. A diagram of the apparatus is shown in Figure 13.

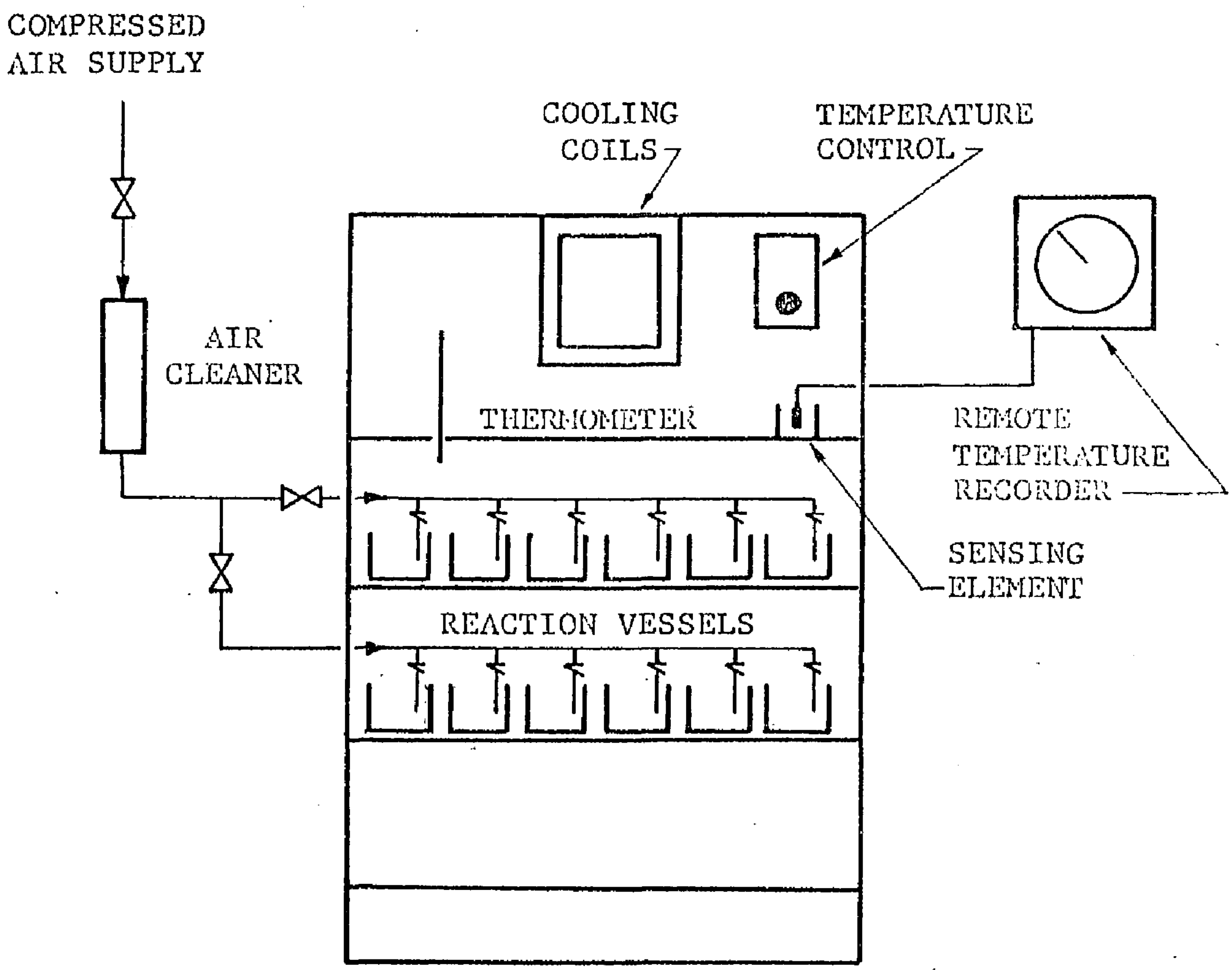
Because of the previous difficulties with temperature control it was decided to conduct a "shake-down" run. In order to evaluate the temperature control, a "Foxboro" temperature recorder of the gas filled bulb type was set up. The temperature sensing bulb was immersed in one of the reactors and the recorder proper was placed outside of the incubator.

Fifteen two-quart Mason jars were utilized as reactors. Nitrogen was added in the form of ammonia to different reactors in four concentrations. In addition, samples were run to which no nitrogen was added. For each nitrogen concentration one reactor was sealed, one open to the atmosphere, and one aerated.

The results of the first run are shown in Table VIII. The temperature recorder drew a smooth concentric circle, indicating that the controller was performing properly. At the end of about 150 hours the reaction had progressed extensively. The aerated samples which had been fortified with various amounts of nitrogen showed the greatest removal of O.C. The use of reactors that were merely open to the air

FIGURE 13

DIAGRAM OF CONSTANT-TEMPERATURE
MULTIPLE REACTOR APPARATUS



FLOW TO INDIVIDUAL REACTORS
ADJUSTED WITH PINCH CLAMPS

TABLE VIII

O.C. IN REACTORS CONTAINING VARIOUS AMOUNTS OF AMMONIA, RUN NO. 1

Filtered Cane Wash Water

Nitrogen Added ppm	Type of Operation	TOTAL HOURS 24	48	77	125	148	172	220	315	340	360	388
None	Open	380	275	318	104	79	39	51	46	59	49	92
None	Aerated		291	352	288	358	110	109	80	84	93	94
None	Sealed		295	292	200	99	100	57	61	62	41	62
33	Open		279	207	134	135	85	68	44	68	71	66
33	Aerated		245	254	100	73	33	23	14	66	31	78
33	Sealed		272	220	212	125	132	78	46	72	71	75
17	Open		228	195	126	129	71	81	38	84	70	68
17	Aerated		245	247	76	16	15	78	15	66	60	67
17	Sealed		245	208	118	99	105	125	40	80	74	65
12	Open		203	202	144	92	65	45	47	72	78	69
12	Aerated		213	248	90	25	17	79	28	82	55	77
12	Sealed		194	210	158	109	155	107	29	76	54	64
10	Open		192	210	160	83	77	92	52	85	92	76
10	Aerated		229	285	116	58	29	93	14	52	60	66
10	Sealed		182	207	171	96	79	99	32	66	72	72

was discontinued and efforts were concentrated on strict aerobic and anaerobic operation.

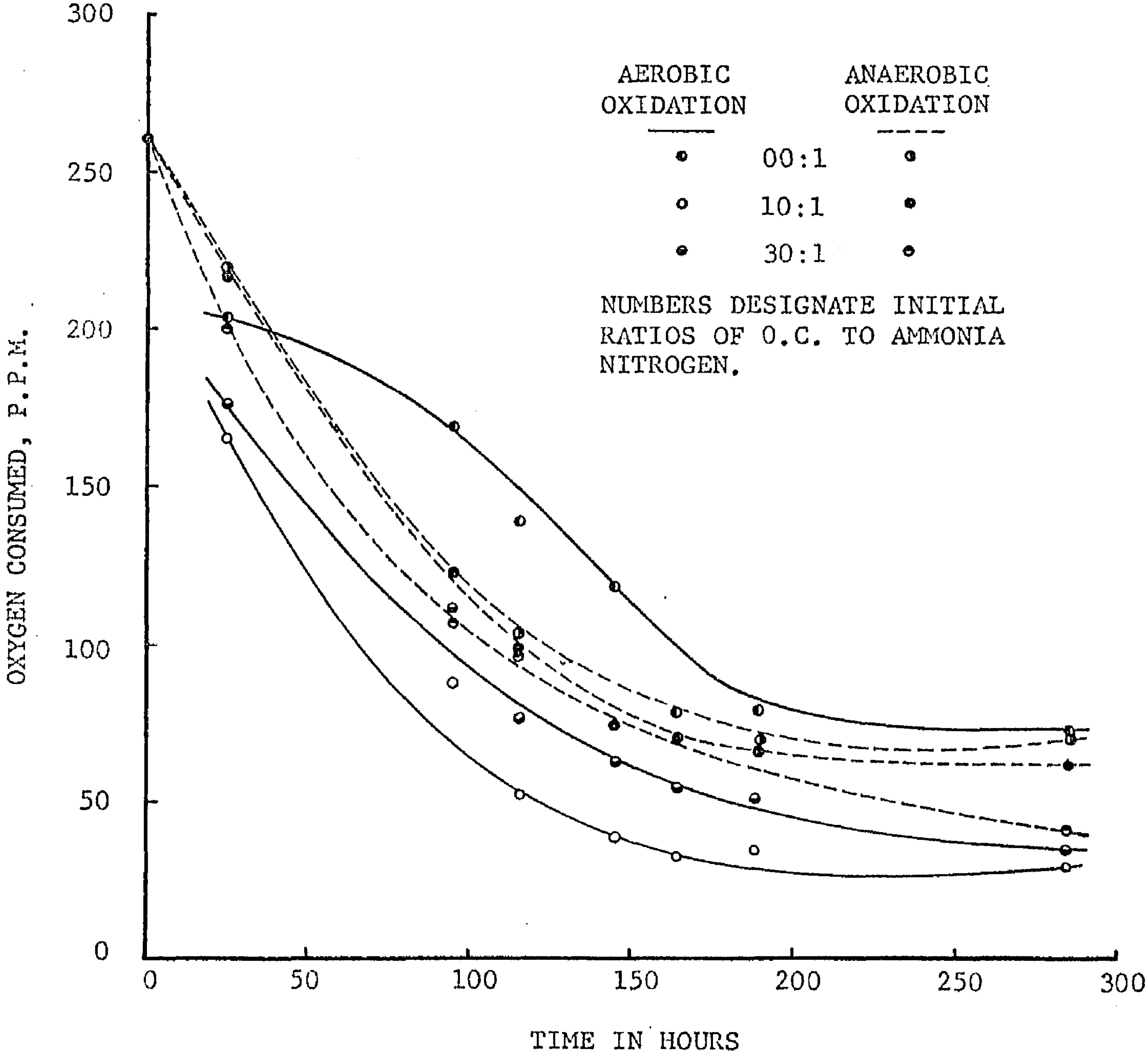
The in-between method of an open beaker was discarded as being too arbitrary with regard to the type of action present. Since a beaker only a few inches deep cannot adequately simulate the true conditions in a pond several feet deep, the exposed surface merely introduces another unknown factor with regard to the D.O. distribution therein.

A second run was made using cane wash water from the frozen storage supply which, as in run No. 1, was first passed through a filter to remove the coarse solids which might tend to decompose during the run and thereby give false results. This filtering was comparable to the preliminary screening and settling necessary for plant treatment of cane wash effluent regardless of the subsequent process. Ten reactors were used. Five were aerated and the rest were sealed to insure anaerobic conditions. This was another "shake-down" run to test the apparatus and no B.O.D. tests were made.

The results of run No. 2 of this series are shown graphically in Figure 14 which is a plot of O.C. vs time for each of the reactors. The initial mixture had an O.C. of 260 ppm and an ammonia nitrogen content of about 2 ppm as determined by a "Taylor Comparator". Nitrogen was added in the form of

FIGURE 14

OXYGEN CONSUMED VERSUS TIME
FOR
FILTERED CANE WASH EFFLUENT
AT 20 °C



ammonia to give nitrogen to O.C. ratios of 0:1, 1:10, 1:20, 1:30, and 1:40 for corresponding anaerobic and aerobic reactors. The temperature was held constant at 20°C. Material from run No. 1 was used as seed.

Aerobic vs Anaerobic Treatment

A look at Figure 14 which shows a family of curves, one for each reactor, illustrates two points of vital importance to this study. The first is that the anaerobic curves tend to be tightly grouped together and are at a higher level than those for the aerobic reactors. The nutrient addition does not show any appreciable effect on the rate of anaerobic reaction. The second is that the aerobic curves are widely spaced, showing a greater amount of stabilization for the reactors having the greatest nitrogen additions.

Nutrient Addition

This run illustrated the importance of proper nutrient addition to optimum biological stabilization of cane wash water. Consequently it was decided to evaluate the effects of adding the key nutrients, nitrogen and phosphorous on the rate of treatment.

Run No. 3 of the series used as feed filtered cane wash water that had been obtained during the previous season. Seed was not added from the previous run, resulting in a rather long "lag" period. The numerical results are shown in Table IX. In the anaerobic reactors there was no B.O.D. reduction for a period of about 200 hours during which the O.C. steadily dropped. At about the time when the B.O.D. had begun to stabilize the O.C. ceased to drop and remained level for the remaining 100 hours in the run. However, in the aerobic reactors both B.O.D. and O.C. dropped off at about the same rate throughout the run. The explanation for this behavior is that under anaerobic conditions the material undergoes certain molecular rearrangements without any lessening in the quantity of organic food potential as expressed by the B.O.D. This is often referred to as the "volatile acids formation" by sewage technologists. It was only after this stage that the B.O.D. began to drop.

With the exception of slight differences of nutrient addition, run No. 4 was similar to run No. 3 and was very carefully carried out to insure accurate results. For the anaerobic reactors there was a very slow rate of stabilization and no similarity between the O.C. and B.O.D. curves. The results of run No. 4 are shown in Table X. Figure 15 shows O.C. and B.O.D. plots for anaerobic and aerobic reactors each of which had a B.O.D./Nitrogen ratio of 10:1 in the initial feed. Although the O.C. curves for both reactors

TABLE IX

O.C. AND B.O.D. IN REACTORS CONTAINING VARIOUS AMOUNTS OF AMMONIA AND PHOSPHOROUS (RUN NO. 3)

Filtered Cane Wash Effluent

T = 20°C

Nitrogen Added ppm		TOTAL HOURS										
		18	40	47	75	114	139	162	210	259	279	306
Anaerobic 0	BOD	269		294				242	208		136	132
	OC				619	515	405	322	355	328	310	330
23	BOD	279		272				252	219		128	107
	OC	532	502	513	584	473	404	398	324	275	271	291
16	BOD	282		279				264	224		120	121
	OC	432	426	497	619	530	414	365	332	280	261	295
13	BOD	288		279				244	216		122	159
	OC	522	472	517	625	556	388	324	341	304	276	294
13*	BOD	262		275				244	220		113	126
	OC	514	408	516	606	550	395	353	342	278	262	297
Aerobic 0	BOD	283		273				180	157		142	62
	OC	534	420	528	565	570	499	443	396	368	320	335
23	BOD	301		278				187	123		123	116
	OC	498	408	514	567	539	470	464	390	340	346	335
16	BOD	274		286				180	133		126	118
	OC	521	438	519	435	511	488	427	387	351	340	312
13	BOD	309		277				165	143		106	100
	OC	534	380	530	576	576	418	358	347	319	314	310
13*	BOD	303		318				176	131		125	116
	OC	524	391	520	532	543	454	380	368	327	301	302

* Includes 6 ppm phosphorous.

TABLE X

O.C. AND B.O.D. IN REACTORS CONTAINING VARIOUS AMOUNTS OF AMMONIA AND PHOSPHOROUS
(RUN NO. 4)

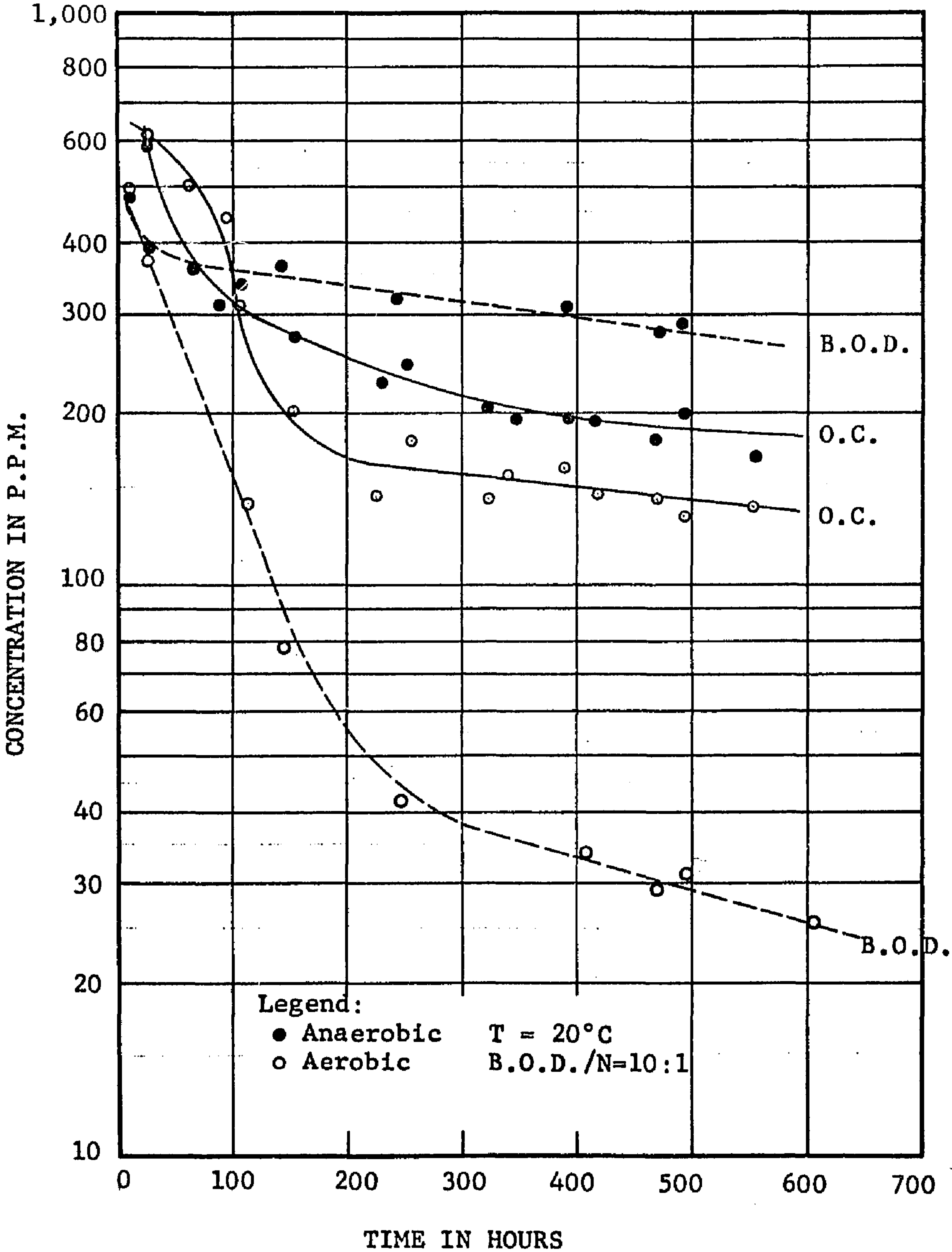
Filtered Cane Wash Effluent

T = 20°C

Nitrogen Added ppm	Phosphorous Added ppm		TOTAL HOURS 27	70	104	132	156	228	252	298	324	394	418
Anaerobic		BOD	372		367								
--	--	OC	613	385	318	-	240	-	252	192	323	230	171
10	--	BOD	372		338								
		OC	584	355	312	-	279	-	243	196	204	-	198
5	--	BOD	363		342								
		OC	609	375	303	226	238	173	-	212	230	222	188
--	3	BOD	375		291								
		OC	605	325	327	203	188	185	236	220	240	194	178
--	6	BOD	377		291								
		OC	610	353	278	212	198	185	-	223	-	229	193
Aerobic		BOD	366		150							29	
--	--	OC	618	525	345	195	173	137	-	139	117	200	142
10	--	BOD	368		135							34	23
		OC	598	500	312	-	200	-	176	115	142	-	144
5	--	BOD	359		95							38	25
		OC	620	468	332	203	175	105	162	95	123	-	122
--	3	BOD	357		94							17	14
		OC	605	505	339	142	130	121	-	102	141	131	132
10	6	BOD	365		98							18	3
		OC	605	475	253	179	129	132	138	110	140	158	104

FIGURE 15

COMPARISON OF B.O.D. AND O.C. CURVES
FOR AEROBIC AND ANAEROBIC REACTORS



were more or less similar there was a striking contrast between the two B.O.D. curves. The plots are semi-logarithmic, therefore the slopes are proportional to the reaction rate constants. This rate of reaction is conveniently expressed by the reaction half lives which are inversely proportional to the reaction rate constants. The half life for the aerobic reactor was 57 hours as compared to 630 hours for the anaerobic one, a ratio of about 12 to 1. This difference agrees with the literature on the relative rates of the two processes and their applications[25]. Generally, the anaerobic process is used at B.O.D. concentrations of several thousand and is usually followed up by aerobic treatment once the B.O.D. is below about 1000 ppm[21].

Because two runs had shown that the aerobic operation was many fold better than anaerobic it was decided to discontinue further anaerobic teşts and concentrate on optimizing the aerobic stabilization of cane wash effluent from the viewpoint of nutrient addition and temperature.

Four additional sets of runs were made. Two were replicas at 20°C and the others were made at 15°C and 24°C. The data from these runs is shown on Tables XI through XV.

<u>Basis for obtaining rate data</u>: The B.O.D.'s from each reactor were plotted (on semi-logarithmic paper) against time.

TABLE XI

SUMMARY OF REACTION HALF-LIVES FOR AEROBIC REACTORS CONTAINING VARIOUS CONCENTRATIONS OF ADDED NUTRIENTS

Temperature: 20°C (Half-Lives in Hours)

Nitrogen Added, ppm	Phosphorous Added, ppm: 0	4.4	8.8
0	24/22	25/22	23/21
19	16/14	16/12	15/12
39	12/14	15/12	15/12

Note: Pairs of numbers are results of replicated runs. Feed material was cane wash effluent having a B.O.D. of 350 ppm.

Source of Variation	Sum of Squares	Degrees of Freedom	Mean Square	F
UPPER NUMBERS				
Between Rows (N)	172.2	2	86.10	59
Between Columns (P)	2.9	2	1.45	1
Residual	5.8	4	1.45	
LOWER NUMBERS (REPLICATE RUN)				
Between Rows (N)	162.0	2	81.0	22.0
Between Columns (P)	4.7	2	2.35	6.5
Residual	1.3	4	0.36	

Standard Deviation between replicates = 2.7. For 2 and 4 degrees of freedom an F of 6.94 is significant at the 5% level and one of 18.0 at the 1% level.

TABLE XII

SUMMARY OF REACTION HALF-LIVES FOR AEROBIC REACTORS CONTAINING VARIOUS CONCENTRATIONS OF ADDED NUTRIENTS

Temperature: 24°C (Half-Lives in Hours)

		Phosphorous Added, ppm		
		0	4.4	8.8
Nitrogen	0	16	14	12
	19	11	11	9
	38	10	9	10

Source of Variation	Sum of Squares	Degrees of Freedom	Mean Square	F
Between Rows (N)	32.6	2	16.3	46.6
Between Columns (P)	6.0	2	3.0	8.6
Residual	1.4	4	0.35	

For 2 and 4 degrees of freedom and F of 6.94 is significant at the 5% level and one of 18.0 at the 1% level.

TABLE XIII

SUMMARY OF REACTION HALF-LIVES FOR AEROBIC REACTORS CONTAINING VARIOUS CONCENTRATIONS OF ADDED NUTRIENTS

Temperature: 15°C (Half-Lives in Hours)

		Phosphorous Added, ppm		
		0	2.2	4.4
Nitrogen Added, ppm	0	34	--	32
	10	--	26	26
	19	21	--	20

TABLE XIV

SUMMARY OF REACTION HALF-LIVES FOR AEROBIC REACTORS CONTAINING VARIOUS CONCENTRATIONS OF ADDED NUTRIENTS

Temperature: 20°C (Half-Lives in Hours)

Using Average Values of 2 Replicated 20°C Runs:

		Phosphorous Added, ppm		
		0	2.2	4.4
Nitrogen Added, ppm	0	23	23.5	22
	10	15	14	13.5
	19	13	13.5	13.5

Source of Variation	Sum of Squares	Degrees of Freedom	Mean Square	F
Between Rows (N)	166.1	2	83.05	207.6
Between Columns (P)	0.9	2	0.45	1.12
Residual	1.6	4	0.40	

TABLE XV

COMPARISON OF THE EFFECTS OF NITROGEN ADDITION AND TEMPERATURE ON REACTION HALF-LIVES FOR AEROBIC STABILIZATION OF CANE WASH EFFLUENT

Temperature °C	Nitrogen Added, ppm* 0.0	4.4	8.8
15	33.0	27.0	21.0
20	22.8	14.1	13.3
24	14.0	11.0	9.5

Source of Variation	Sum of Squares	Degrees of Freedom	Mean Square	F
Between Rows (T)	373.0	2	186.5	36.3
Between Columns (N)	117.6	2	58.8	11.5
Residual	20.5	4	5.13	

For 2 and 4 degrees of freedom an F of 6.94 is significane at the 5% level and one of 18.0 at the 1% level.

* Numbers read from smoothed curves shown on graph, therefore are slightly different from those shown on other tables.

The best line was drawn through the maximum slope and from it a half-life for each reactor was obtained.

If c = concentration

t = time

and k = reaction rate constant

then the basis of this method is as follows:

$$-\frac{dc}{dt} = kc$$

$$-\frac{dc}{c} = kdt$$

(the minus sign indicates that the concentration is decreasing)

$$-\ln c = kt + c$$

$$-[\ln c_2 - \ln c_1] = k\,(t_2 - t_1)$$

$$k = \frac{\ln\,(c_1/c_2)}{t_2 - t_1}$$

Therefore, a semi-log plot of C (B.O.D.) against time will yeild a straight line having a slope equal to k.

The actual measurements of k from reaction data may be simplified by using the half-life, i.e., the time required for the concentration (B.O.D.) to drop in half.

Since $\ln \frac{2}{1} = +0.693$

then $k = \dfrac{+0.693}{t\,\frac{1}{2}}$

This procedure was followed to obtain the half-life values shown in Tables XI, XII, XIII, XIV, and XV. Note that the values in Table XI were replicated by making two runs under identical conditions. The feed material for all runs was the same, consisting of cane wash effluent which had been well mixed in a large vat, filtered and then frozen at -20°F in five gallon cans. This procedure assured a uniform feed for all runs.

Quantitative Evaluation of Kinetic Data

Each run was made using different concentrations of nitrogen and phosphate nutrients. The nitrogen was supplied in the form of ammonia and the phosphorous as monobasic potassium phosphate (KH_2PO_4).

The statistical data relating to the significance of each are shown on the tables in which the half-lives are listed.

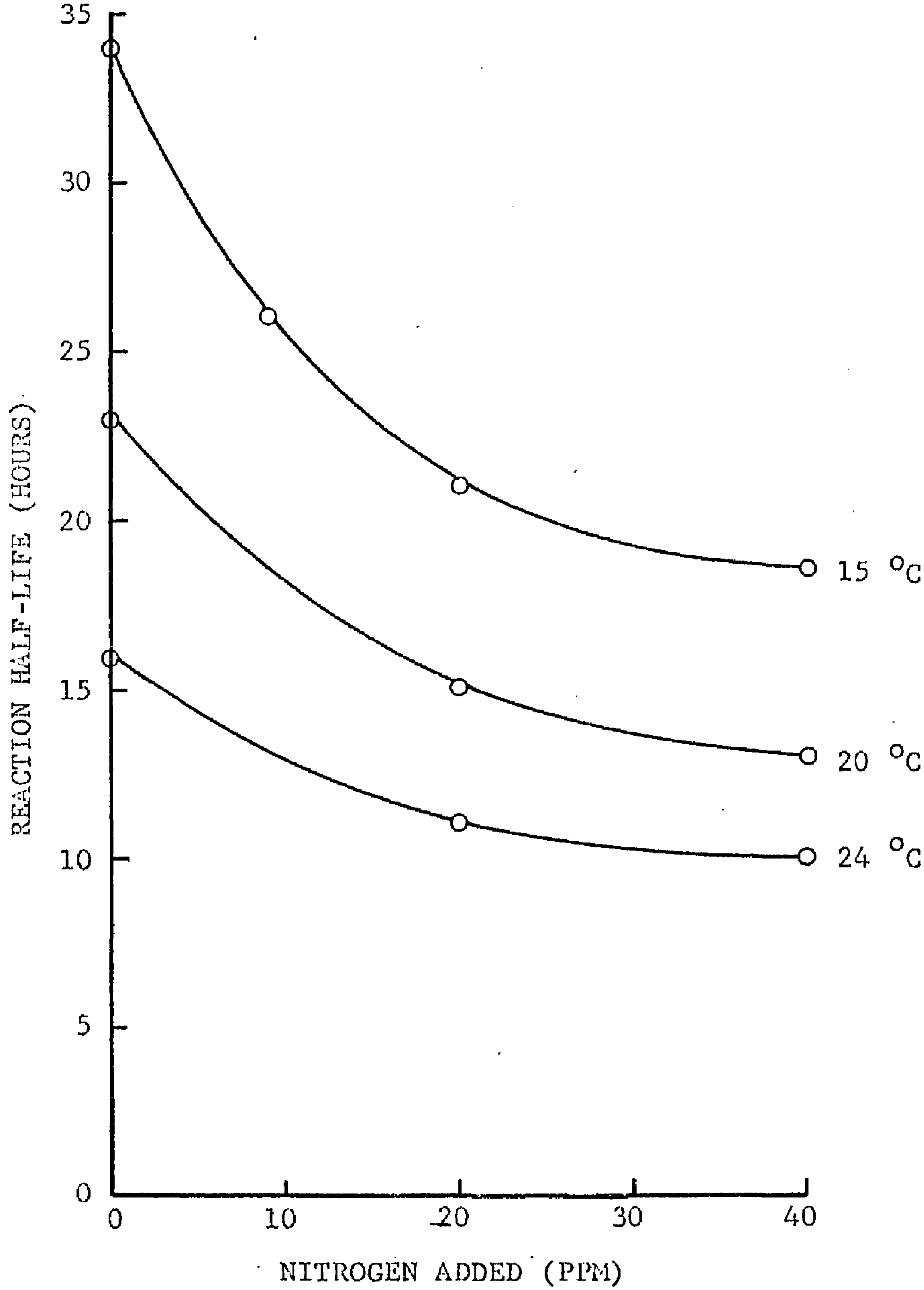
Effect of Nutrient Addition

An analysis of variance was made on each set of runs to determine how significant the variables were insofar as their influence upon the reaction half-lives. It was found that phosphorous was not significant at the 5% confidence level. Nitrogen and temperature were both of significance at better than the 1% level.

A plot of half-life versus nitrogen addition at the three temperatures investigated is shown in Figure 16. It can be seen that the slope of each of the lines drops off rapidly

FIGURE 16

EFFECT OF NITROGEN ON REACTION HALF-LIVES
OF
CANE WASH EFFLUENT



with increasing nitrogen addition, approaching the horizontal at about 40 ppm. The optimum nitrogen dosage lies between 15-25 ppm depending upon economic factors. A good approximation would be to use a ratio of 1:20 nitrogen to B.O.D. as a basis for commercial design. Then if the installation become over-loaded an increased nitrogen addition would maintain a satisfactory effluent, but at a price.

Effect of temperature: The rates of most chemical reactions increase greatly with temperature. A commonly used approximation, attributed to van't Hoff is that the rate doubles for each 10°C increase in temperature. Arrhenius (1889) set forth the following relation between equilibrium constant with temperature.

$$\frac{d\ (\ln Kc)}{dt} = \frac{\Delta E}{RT^2}$$

Since the equilibrium constant (K) is merely the ratio of rate constant (k) for forward and reverse reactions (law of mass-action) the variation of rate constant with temperature is given by the equation below.

$$\frac{d\ (\ln k)}{dt} = \frac{\Delta E}{RT^2}\text{(activation)}$$

This can be resolved into the useable form:

$$\frac{t}{t_o} = e^{c(T_o - T)} = \theta^{(T_o - T)}$$

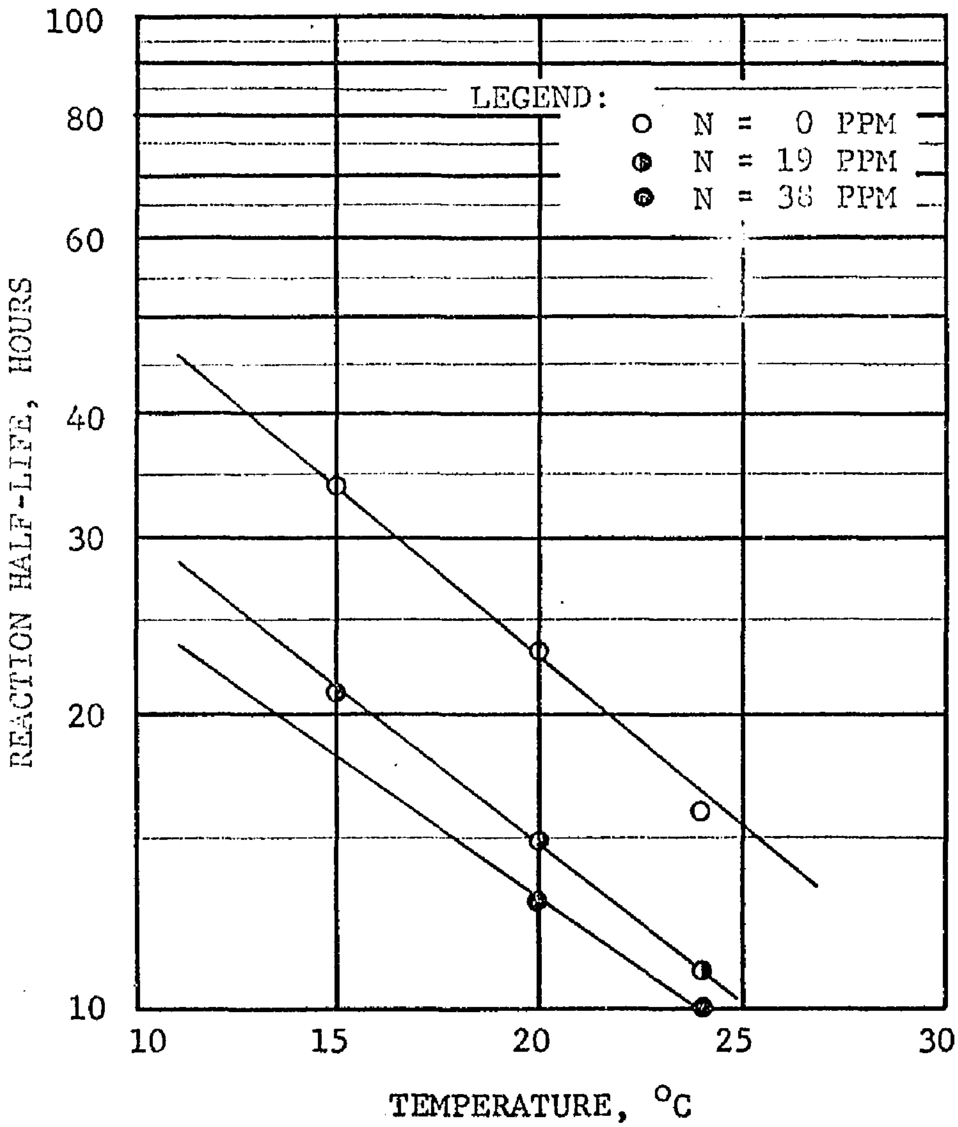
t = reaction time

T = temperature

Figure 17 is a semi-logarithmic plot of half-life versus temperature for the three levels of nitrogen addition. This plot gives essentially a straight line having a negative slope. From the slope it is possible to evaluate the term θ. For this application it was found that θ = 1.08, which fit very nearly to the van't Hoff-Arrhenius rule of thumb that the rate is halved for each 10°C decrease in temperature[13]. For all practical purposes this rule may be applied to the rate of bio-stabilization of cane wash effluent.

FIGURE 17

EFFECT OF TEMPERATURE ON REACTION HALF-LIVES
OF
CANE WASH EFFLUENT



CHAPTER VII

CONCLUSIONS

From a study of eleven commercial raw sugar factories in Louisiana it was found that:

1. The water pollution by these factories results from organic enrichment of the stream.

2. There are six general effluents: (1) condenser cooling water, (2) cane wash water, (3) soda and acid, (4) filter cake, (5) floor sweepings, and (6) bearing cooling water. In a well operated factory only cane wash water and condenser cooling water present serious pollution problems.

3. With a properly designed, maintained and operated factory the condenser cooling water is sufficiently free of organic pollutional material to enter a public stream. It should first be passed through a spray pond in order to cool it and add oxygen so that no damage will be done to the stream from oxygen depletion by the warm deaerated effluent. This will aid in self-purification to remove any small amounts of organic material entrained in the cooling water in the barometric condensers.

4. Cane wash water, because of its high B.O.D. and suspended solids content, must not be allowed to enter public waters. Most of the solids can be removed by simple settling,

but the dissolved B.O.D. remains high enough to make it intolerable to the stream.

5. Most factories do not have, and cannot afford, facilities to impound all of the cane wash water they need to use for the period of time required for it to stabilize naturally. By using two stages of wash water re-cycle during the cane washing operation it has been found possible to reduce the volume of effluent to at least one-third of the original. This reduces the size of treatment facilities needed and results in a more concentrated effluent which, for the same total B.O.D., may be treated more rapidly.

6. No ill effects upon factory operation have resulted because of cane wash water re-cycle. The circulating systems have remained free of biological growths. If slimes should build up in the wash water system they could be easily controlled by infrequent "shock" chlorination carried out by dumping chloride of lime (H.T.H.) into the recirculating water pump.

7. Pilot plant reaction studies have shown that settled liquid cane wash effluent can be stabilized by aerobic oxidation ponds. Anaerobic oxidation requires many times the aerobic treatment period. At an average water temperature of 20°C, which is typical of commercial conditions, the

effluent can be stabilized aerobically at a rate corresponding to first order reaction half-life of 15 hours when 20 ppm of nitrogen are added as nutrient. Greater additions of nitrogen are not considered economic. Potassium and phosphorous addition did not significantly speed-up the rate of stabilization. The rate of the stabilization follows the van't Hoff-Arrhenius relation and consequently is doubled for each 10°C increase in temperature. The total holdup time for a properly operated treatment installation should not exceed 70 hours. Based on the average flow of 1000 GPM of cane wash effluent for all Louisiana factories surveyed, a pond area of about 4 acres should be adequate. This amount of land is available to practically every factory. In addition, the volume of effluent to be treated can be reduced to one-third by the use of recycle washing. This would further reduce the land requirement.

8. The O.C. is not representative of the B.O.D. on an absolute numerical basis for an isolated single effluent sample. However, for liquid effluent the difference in B.O.D. and O.C. of a stream passing through a sugar mill can be related by the equation:

$$\Delta \text{ O.C.} = 0.9 \, \Delta \text{ B.O.D.}$$

Where Δ designates the increase resulting in a given stream passing through the factory.

9. The α-naphthol test for dissolved sugars was found to have a maximum sensitivity of 10 ppm when fresh solutions were used laboratory conditions. In several cases factory α-naphthol tests detected no sugars where there was a B.O.D. pick-up of as high as 50 ppm. This lack of sensitivity was traced to the use of old reagent solutions and inadequate lighting.

10. Based on current conditions the savings resulting from pollution abatement through increased sugar yields and better operation permissible in the case of cane washing will rapidly pay-out the costs incurred for repairs and the installation of additional equipment. In some of the operating factories studied the savings from less than one week of improved operation paid these costs.

11. Pollution abatement is a necessity if the Louisiana Sugar Industry is to continue in our present day society.

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A P P E N D I X

APPENDIX I

NOMENCLATURE

B.O.D.	-	Biochemical Oxygen Demand
c	-	A constant
D.O.	-	Dissolved Oxygen
E	-	Internal energy
GPM	-	Gallons per minute
K	-	Equilibrium constant
k	-	Reaction rate constant
ml	-	Milliliters
O.C.	-	Oxygen Consumed from Permanganate
ppm	-	Parts per million
R	-	Universal gas constant
T	-	Temperature
t	-	Time
θ	-	A constant

APPENDIX II

EXPLANATION OF TERMS AND ANALYTICAL TESTS

Dissolved Oxygen (D.O.): Adequate dissolved oxygen is necessary for the life of fish and other aquatic organisms. Its concentration also indicates corrosivity of water, photosynthetic activity, septicity, etc. This test indicates the condition of the water at the time the sample was obtained. It is primarily a field method for determining the amount of oxygen available for the decomposition process if taken above the point of discharge and the residual oxygen during the active biological decomposition if taken below the point of discharge. Since the amount of oxygen that can be dissolved in water varies with temperature, the temperature of the sample should be taken.

The dissolved oxygen test is performed by a modification of the "Winkler" method. From the level of the dissolved oxygen only it indicates whether the supply of oxygen is greater than the demand of biological decomposition of the waste in the stream, and does not measure the total oxygen demand of the effluent. If the dissolved oxygen content does not drop below four parts per million by weight there is little danger to aquatic life, providing the waste contains no poisonous material. Coogan and Biglane[3] state that a D.O.

of at least 5 parts per million (ppm) should be maintained in a stream.

The procedures used in performing this and other tests used in this project are described in detail in "Standard Methods for the Examination of Water, Sewage, and Industrial Wastes"[1].

Biochemical Oxygen Demand (B.O.D.): In the study of organic pollution it is important to know how much total oxygen will be required to completely stabilize the waste. This information is provided by a determination known as the Biochemical Oxygen Demand, or, more commonly, B.O.D. test. The B.O.D. represents the oxygen used by the material in the course of its complete biochemical stabilization. It is not always related to the oxygen requirement for complete chemical combustion, but is a function of the usability of the material as a bacterial food, and the amount of oxygen used by the bacteria during the bio-oxidation.

Complete stabilization requires more than 100 days at 20°C used in the test, but such long periods of incubation are impracticable in most cases. Consequently, a 5 day period is used. The test consists of determining the dissolved oxygen of samples of a given effluent before and after the incubation period. In the case of highly polluted effluents, dilution of the waste is made to prevent complete removal of the oxygen from the incubated samples. The loss of oxygen is the biochemical oxygen demand and is expressed in parts per million by weight. All B.O.D. determinations in this program are the standard 5 day, 20°C B.O.D.

Oxygen Consumed (O.C.): Oxygen consumed from permanganate is that amount of oxygen removed from potassium permanganate by a water sample when it is digested for 30 minutes in a boiling water bath with a definite strength of permanganate and dilute sulfuric acid. The carbon, not the nitrogen, in organic matter is oxidized by potassium permanganate. Because of this the O.C. is sometimes erroneously considered as indicating the amount of carbonaceous matter present. However, the determination indicates only a part of the carbon. This is because the carbon in nitrogenous material is not readily oxidized as is carbon in carbonaceous organic matter. It also does not differentiate directly the carbon present in useable form from that in stable organic compounds.

The usefulness of this test lies in that it can be made with relatively crude equipment by semi-skilled personnel. It will be shown later that the O.C. gives a good indication of the B.O.D. for most sugar factory effluents and is closely related when differences due to added organic material are being evaluated. For a known type of sugar factory effluent the O.C. has proven satisfactory to evaluate B.O.D. added using the correlation developed during the research program. The O.C. is particularly useful for routine monitoring of cooling water streams because it can be run by the factory laboratory on a round-the-clock basis. The pollutional material that enters the stream is principally a mixture of sugars, a type of material having a B.O.D. and O.C. which show a close relationship.

At best, the O.C. is a field approximation of the B.O.D. for it is the B.O.D. which is the true index of the pollutional quality of an organic waste.

APPENDIX III

SUITABILITY OF THE O.C. TEST AS A MEASURE OF B.O.D.

It was briefly mentioned in a previous section that the Oxygen Consumed From Permanganate and the Biochemical Oxygen Demand, although roughly equivalent for readily oxidizable material, did not have the same numerical values in all cases. This is because the O.C. does not differentiate between stable and unstable organic material. The carbon in nitrogen-containing material is not so readily oxidized as is the carbon in carbonaceous organic material. The O.C. test suffers from the disadvantage that it is sensitive to many inorganic compounds which reduce permanganate but are inert biochemically.

The O.C. test can be run rather quickly in the field by semi-skilled workers and with a minimum of equipment. The B.O.D., on the other hand, requires a constant temperature incubator, numerous specially made bottles, aerated dilution water fortified with proper nutrients and require 5 days for completion. It was advantageous to the project to have the mill personnel run only the O.C. and attempt to correlate this with the B.O.D. In order to do this a large number of tests were duplicated. That is, both an O.C. and a B.O.D. were run on each of numerous effluent samples.

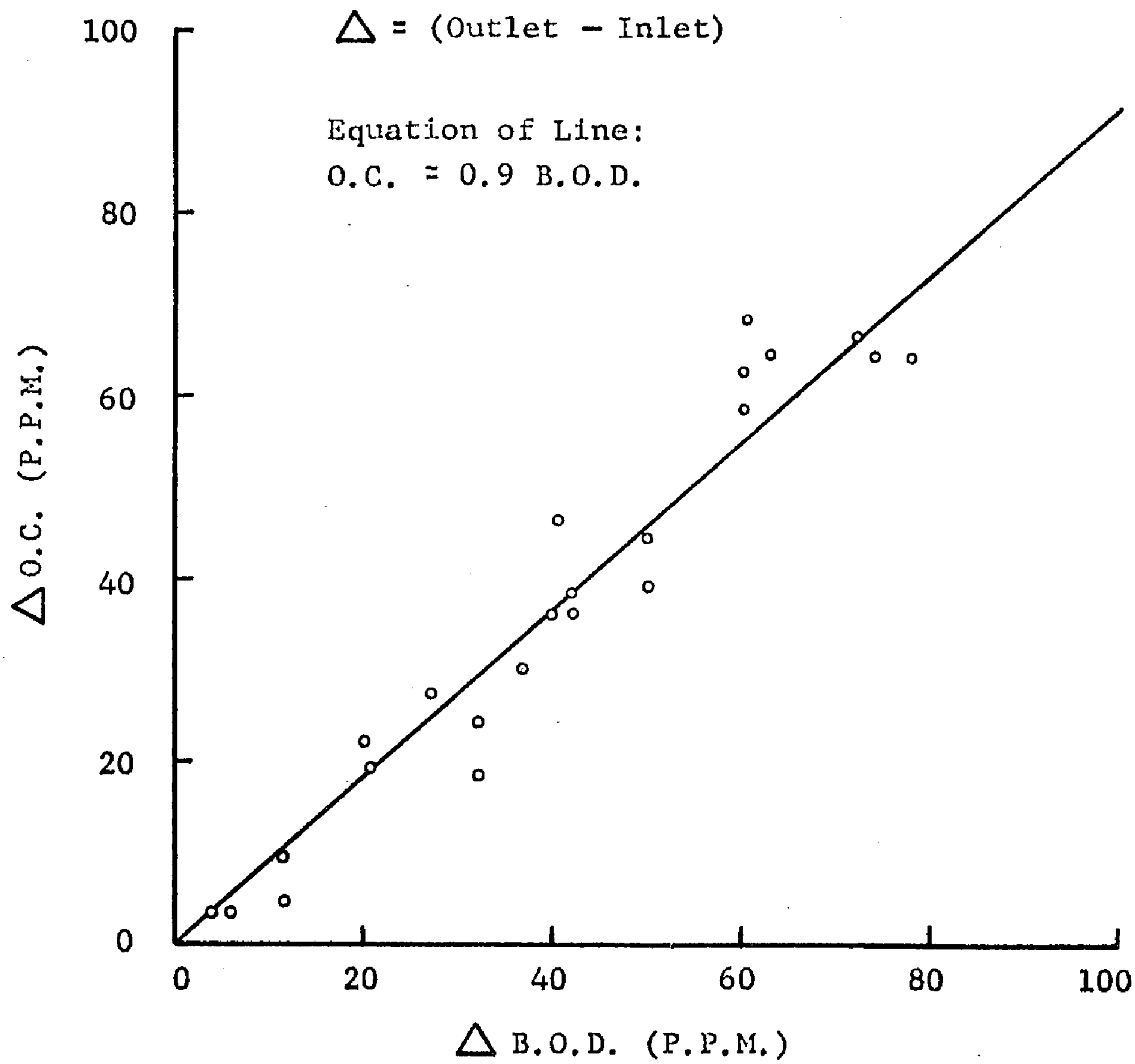
The relationship between B.O.D. and O.C. is shown in

Figure 18. It was found, as might be expected from the previous discussions of the tests, that there was no absolute relation between B.O.D. and O.C. on straight effluent samples. However, due to the fact that the pollutional matter from sugar mills is of readily oxidizable nature, both chemically and biologically, a fairly good relation was obtained between the differences shown by a stream entering and then leaving a factory. In other words, although the material entering the factory did not have a B.O.D. and O.C. that were anywhere similar, the material added to it had values of the two that were nearly identical. Because of this, the differences found leaving minus entering were nearly the same.

It must be remembered that the tests are run on diluted samples and the numerical results are therefore very sensitive to errors in the titration step involved in each. According to "Standard Methods"[1], the O.C. has a standard deviation of 0.08 ml for sugars. Both of these tests were run on the basis of a maximum of 10 ml requirement for the undiluted effluent. Therefore, the expected precision should be about 0.15 multiplied by the ratio of diluted to undiluted sample in the actual test. Generally, this should be about $\pm$ 3 per cent of the value since dilutions are usually made to give a mid-range reading on the burette. In addition to

FIGURE 18

CORRELATION OF O.C. WITH B.O.D.
FOR LIQUID SUGAR FACTORY EFFLUENTS



errors in titration there are errors in measurement and in digestion which cause the σ (standard deviation) to vary a total of about $\pm$ 13 per cent for the run of the mill sample.

The relation obtained by a "least squares" regression of the data available indicated that the best relation for sugar mill effluents is O.C. is equal to 0.9 B.O.D.

Figure 18 shows the mill relation of O.C. to B.O.D. for general liquid sugar factory effluent.

APPENDIX IV

THE α-NAPHTHOL TEST FOR DISSOLVED SUGARS

From the study made it can be concluded that the sensitivity of the α-Naphthol solution of 5% strength is 10 ppm for a solution not more than one month old when the following procedure is used in sample analysis.

1. Rinse the test tube with the solution to be tested.
2. Pour in about 1-2 inches of sample.
3. Add enough α-Naphthol, usually about 8-12 drops, to give a faint cloudy solution. If the solution clouds up completely, start over.
4. Add concentrated sulfuric acid by allowing it to run down the side of the test tube until a 1/2-3/4 inch layer is formed. Gently swirl or roll the solution, but do not let the layers mix. Repeat this swirling 2 or 3 times over the interval of 2-3 minutes. If sugar is present a band of color will appear between the layers. This band will be purple to light maroon, depending on the concentration. The light maroon color is indicative of about 10 ppm sugar.

It may be further concluded for the same concentration sugar samples that the α-Naphthol solution stored in dark bottles will give better reproducibility than that stored in light bottles. This is especially true toward the end of the month.

Finally, from the results it may be concluded that unless the proper light is present, the detectable sensitivity may drop as much as 3 ppm. The best light found in this study is the yellow fluorescent lamp.

APPENDIX V

TABULATED CANE WASH

CLARIFICATION TEST RESULTS

DATA—CANE WASH WATER—NEW IBERIA CO-OPERATIVE, INC.

Sample No.	1	2	3	4
Alum, ppm	100	150	200	250
Aid, ppm	---	---	---	---
Floc	V. Good	V. Good	V. Good	V. Good
Supernatant	Clear	Clear	Clear	Clear
Color	Little	Little	Little	Little

NOTE: Test 1 (1st Sample): This was the first sample of CW water collected by the Lab. boy supposedly from under the cane table. The water seemed remarkably clean to start with.

Alum, ppm	100	150	200	250
Aid, ppm (Seperan)	1	1	1	1
Floc	Slightly larger floc than in test 1.			
Supernatant	Much the same as above. Maybe a slight improvement.			
Color				

NOTE: Test 2 (1st Sample): Same water sample as was used in test 1.

Samples were slow-mixed for 11 minutes and allowed to settle for 20 minutes. Tests were run in 1 liter beakers.

Sample No.	1	2	3	4	5
Lime, ppm	200	300	400	500	700
Floc	None	None	V. Small	V. Small	Small
Supernatant	Opaque	Opaque	Opaque	Opaque	Opaque
Color	---	---	---	---	---

NOTE: Test 3 (a second water sample): Most of the floc particles settled in about 5 min. However, supernatant remained opaque as floc was not complete, indicating too small dosage. The addition of excess lime to the hand-stirred beaker produced a good floc which settled fast leaving a clear supernatant. However, the color of the supernatant was high.

Sample No.	1	2	3	4
Lime	200	300	400	500
Aid, Seperan 2610	5 ppm	5 ppm	5 ppm	5 ppm
Floc	Practically None	Small Little	Small Little	Floc wore this without aid in test 3
Supernatant	Opaque	Opaque	Opaque	Opaque
Color	---	---	---	---

NOTE: Test 4 (2nd water sample)

DATA—CANE WASH WATER—NEW IBERIA CO-OPERATIVE, INC. (Cont.)

Sample No.	1	2	3	4
Lime	200	200	400	500
Iron	200	200	200	200
Floc	Poor	Medium	Good	V. Good
Supernatant	Opaque	Straggle	Clear	Clear
Color	---	Red	Red	Red
NOTE: Test 5 (2nd water sample)				
Lime	200	300	400	500
Iron	200	200	200	200
Aid (Seperan 2610)	5	5	5	5
Floc	Medium	Med. Good	Good	V. Good
Supernatant	Opaque	Straggle	Clear	Clear
Color	---	---	Red	Red
NOTE: Test 6 (2nd water sample): Floc seemed notably larger with the aid, but did not seem to settle much faster. Most of the floc that formed seemed to settle within 5 minutes.				
Alum	25	50	75	100
Floc	None	None	None	None
Color	---	---	---	---
Supernatant	Opaque	Opaque	Opaque	Opaque
NOTE: Test 7 (3rd water sample): Dose obviously too mild.				
Alum	125	150	175	200
Floc	None	V. Small	Small	Small-Med.
Color	---	---	---	---
Supernatant	Opaque	Opaque	Opaque	Opaque
NOTE: Test 8 (3rd water sample): It appears that 200 ppm is less than the required dose for good flocculation as was found with the Sterling CW water				
Alum	25	50	75	100
Aid (Seperan 2610)	5	5	5	5
Floc	None	None	None	None
Color	---	---	---	---
Supernatant	Opaque	Opaque	Opaque	Opaque
NOTE: Test 9 (3rd water sample): Dose obviously too small.				
Alum	125	150	175	200
Aid (Seperan 2610)	5	5	5	5
Floc	None	V. Small	Small	Medium
Color	---	---	---	---
Supernatant	Opaque	Opaque	Opaque	Many Straggles
NOTE: Test 10 (3rd water sample): Very little improvement could be noticed by the addition of the aid.				

DATA—CANE WASH WATER—NEW IBERIA CO-OPERATIVE, INC. (Cont.)

Sample No.	1	2	3	4	5
Aid, 1 ppm	Seperon 2610	Aerofloc 548	Aerofloc 552	Floxel	None
Floc	None	None	None	None	None
Supernatant	Opaque	Opaque	Opaque	Opaque	Opaque
Color	---	---	---	---	---
NOTE: Test 11 (4th water sample)					
Aid, 50 ppm	Seperon 2610	Aerofloc 548	Aerofloc 552	Floxel	None
Floc	None	None	None	None	None
Supernatant	Opaque	Opaque	Opaque	Opaque	Opaque
Color	---	---	---	---	---
NOTE: Test 12 (4th water sample): Both these tests showed no improvement by the addition of the aid.					
Aid, 5 ppm	S-3000	S-3019	S-3059	None	
Floc	None	None	None	None	
Supernatant	Opaque	Opaque	Opaque	Opaque	
NOTE: Test 13 (4th water sample): Only the sand, etc. settled to the bottom—most of which settled within the 1st five minutes.					
Aid, 10 ppm	S-3000	S-3019	S-3059	None	
Floc	None	None	None	None	
Supernatant	Opaque	Opaque	Opaque	Opaque	
NOTE: Test 14 (4th water sample)					
Aid, 50 ppm	S-3000	S-3019	S-3059	None	
Floc	None	None	None	None	
Supernatant	Opaque	Opaque	Opaque	Opaque	
NOTE: Test 15 (4th water sample)					
Alum	100	200	300	400	
Floc	None	Medium	Good	Good	
Supernatant	Opaque	Straggles	Clear	Clear	
Color	---	Slight	Slight	Slight	
NOTE: Test 16 (5th water sample)					
Alum	100	200	300	400	
Aid (Seperon 2610)	50	50	50	50	
Floc	None	Good	Good	Good	
Supernatant	Opaque	Clear	Clear	Clear	
Color	---	Slight	Slight	Slight	
NOTE: Test 17 (5th water sample): Could be an improvement due to the aid. Should have tried at smaller dose as 50 ppm is impracticable. However, even at 50 ppm improvement doesn't seem sufficient to warrant the use of aid.					

DATA—CANE WASH WATER—NEW IBERIA CO-OPERATIVE, INC. (Cont.)

Sample No.	1	2	3	4	5
Lime	200	400	600	800	
Aid, 5 ppm	S-3000	S-3000	S-3000	S-3000	
Floc	V.Small Little	Small Little	Medium Little	Large, but evidently not enought for complete clarification	
Supernatant	Opaque	Opaque	Opaque	Opaque	
Color	---	---	reddish	reddish	
NOTE: Test 18 (6th water sample)					
Lime	200	400	600	800	
Iron	100	100	100	100	
Aid, 5 ppm	S-3000	S-3000	S-3000	S-3000	
Floc	V.Small Little	Small	Fiar	V.Good	
Supernatant	Opaque	Opaque	Clear	Clear	
Color	---	reddish	reddish	reddish	
NOTE: Test 19 (6th water sample)					
Lime	200	200	200	200	
Aid, 1 ppm	Sep-2610	AF-548	AF-552	S-3000	
Floc	Little	Little	Little	Medium	
Supernatant	Z	Y	X	B	
NOTE: Test 20 (6th water sample): The letters represent the quality of the supernatant					
Sample No.	5	6	7	8	
Lime	200	200	200	200	
Aid, 1 ppm	S-3019	S-3059	Floxel	None	
Floc	Medium	Medium	Medium	V.Little	
Supernatant	Opaque	Opaque	Opaque	Opaque	
	C	D	A	---	
NOTE: A, B, C and D were very much better than X, Y and Z.					

DATA—CANE WASH WATER, STERLING SUGARS
11/16/55
Raw Water: pH = 7.2; Temp.: Variable

Sample No.	1	2	3	4	Note
		Run No. 1			
Alum, ppm	20	50	80	110	Turbidity too
Lime, ppm	--	--	--	---	high for visi-
Iron, ppm	--	--	--	---	bility of
Floc	None	None	None	None	floc
		Run No. 2			
Alum, ppm	--	--	Coagulent Aid, Hagen and		
Lime, ppm	240	240	Floxel Added—no improvement—		
Iron, ppm	90	60	water was very red on long		
pH	7.1	7.4	settling.		
Floc	Poor	Poor			
		Run No. 3			
Alum, ppm	--	--	--	--	Floc was very
Lime, ppm	→ 240	→ 240	→ 300	500	fine on all
Iron, ppm	120	160	90	90 →	samples. De-
pH	→ 9.8	9.7	10.7	11.5	cane water was
Floc	Poor	Fair	Fair	Fair	very red
		Run No. 4			
Alum, ppm	→ 80	100	120	160	
Lime, ppm	→ 250	→ 250	→ 250	→ 250	
Iron, ppm	→ --	--	--	--	
pH	9.4	10.2	10.1	9.8	
Floc	Poor	Poor	Poor	Poor	
		Run No. 5			
Alum, ppm	→ 80	80	80	80	
Lime, ppm	→ 250	→ 200	→ 150	→ 100	
Iron, ppm	→ --	--	--	--	
pH	→ 10.0	9.7	9.4	9.1	
Floc	Poor	Poor	None	None	

DATA—CANE WASH WATER, STERLING SUGARS
11/16/55
Hand Stirred Sample: 220 ppm Alum
pH = 5.7
Good Floc and Clarification

Sample No.	1	2	3	4	Note
		Run No. 1			
Alum →	220	180			
pH →	---	---			
Floc	Fair	Poor			
		Run No. 2			
Alum	150	200	220	250	11 Min. Stir-
pH	6.2	5.8	5.6	5.4	ring at 60 RPM
Floc	Poor	Fair	Fair	Good	
		Run No. 3			
Alum →	200	150	100	---	
Iron →	---	50	100	200	
Lime →	---	---	---	200	
pH →	5.8	---	---	9.1	
Floc →	Fair	Poor	None	Settling with red water	
		Run No. 4			
Iron →	---	30	---	---	
Alum →	120	60	180	90	
Lime →	---	250	---	---	
Aid, ppm →	#7(1.0)	Floxel	#7(1.0)	#7(1.0)	
pH	6.2	9.8	6.2	6.5	
Floc	Poor	Poor	Poor	Poor	

Sample No.	1	2	3	4	5	6
			Run No. 5			
Lime	200	300	400	500	600	700
pH	10.4	10.9	11.2	11.4	11.6	11.8
Floc	None	Tinge	Poor	Fair	Fair	Fair
Supernatant	Opaque	Tur.	Tur.	SE	SE =	SE
Color			High	High	High	High

Run No. 6

Same as Above + 4 drops of Floxel/L	
Flox	Results similar to above except slightly better.
Supernatant	NOTE: 600 ppm lime, not as good as
Color	220 ppm Alum.

CANE WASH EFFLUENT (Jar Test Results)
Run: Oct. 28, 1955

Run No.	Mud in 10 Total Min.	Alum ppm	pH	Lime ppm	Remarks
1	.375/7	188	86	8.9	Small floc-cloudy supernatant
2	.375/7	222	110	9.1	Size of floc increased with
3	.375/7	256	120	9.0	increase in chem. dosage
4	.375/7	290	135	9.0	Reasonable floc-supernatant
5	.5/7	325	155	8.5	clear but many stragglers
6					Most of floc settled
7					within 5 minutes
8					
9					
10					

Run No.	Alum	Lime	pH	Remarks
1	17	17	8.8	No floc
2	34	31	9.2	No floc
3	51	17	8.6	No floc
4	68	20	8.6	No floc
5	86	26	8.5	No floc
6	103	38	8.6	No floc
7	124	48	8.7	Slight floc--Opaque Supernatant
8	142	43	8.7	Slight floc--Opaque Supernatant
9	159	46	8.5	Slight floc--Opaque Supernatant
10	171	57	8.5	Much better floc but opaque supernatant

CANE WASH EFFLUENTS (Jar Test Results)
Run: Oct. 28, 1955

Note: These tests were conducted in 7" x 1" test tubes.

Run No.	Mud in 10 Total min.	Alum ppm	pH	Lime ppm	Remarks
1	.375/7	188	6.5	26	little floc little settling-Supernatant Opaque
2	.375/7	188	7.0	34	little floc little settling-Supernatant Opaque
3	.375/7	188	7.5	43	little floc little settling-Supernatant Opaque
4	.375/7	188	8.0	51	little floc little settling-Supernatant Opaque
5	.375/7	188	8.5	77	little floc little settling-Supernatant Opaque
6	.375/7	188	9.0	105	little floc little settling-Supernatant Opaque
7	.375/7	188	9.5	120	little floc little settling-Supernatant Opaque
8	.375/7	188	10.0	150	little floc little settling-Supernatant Opaque
9	.375/7	188	10.5	170	reasonable settling and floc-Supernatant Opaque
10	.375/7	188	11.0	200	reasonable settling and floc-Supernatant Opaque
11	.5/7	220	10.6	230	Good settling, clear brown supernatant, many straggles

Initial pH of water 7.4

All samples had much carbon floating on the surface

The above run was conducted at varying pH. The pH being varied by the addition of lime.

All samples were strained free of large bagacillo particles through a juice strainer.

VITA

Joseph Edward Wheeler, Jr. was born at Saint Louis, Missouri, on June 10, 1932. He attended schools at Augusta, Maine; Memphis, Tennessee; and Alexandria, Louisiana, and was graduated from Menard Memorial High School in May, 1950.

He entered Louisiana State University in September, 1950 receiving a Bachelors Degree in Chemical Engineering in May, 1954, and a Masters Degree in Chemical Engineering in May, 1956.

From 1954 until 1958, he held the position of Research Fellow in the Department of Chemical Engineering at Louisiana State University and has also worked in his present position as a Technologist for Shell Oil Company for slightly over two years, including summer employment.

He is presently a candidate for the Doctor of Philosophy Degree in Chemical Engineering.

EXAMINATION AND THESIS REPORT

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Major Field: Chemical Engineering

Title of Thesis: An Engineering Study of the Effluent Disposal Problems of the Louisiana Raw Sugar Industry

Approved:

Jesse Coates
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Date of Examination:

July 28, 1959